Received: 1 November 2021 | Revised: 17 December 2021 | Accepted: 19 December 2021

DOI: 10.1002/bkcs.12462

COMMUNICATION

BKCS BULLETIN OF THE KOREAN CHEMICAL SOCIETY

Deterministic transfer of thin carbon nanotube film

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Funding information
Ministry of Education, Science, and Technology, Grant/Award Numbers: 2020R1A4A1017737, 2020R1F1A1076983

Abstract

Although successfully implemented for optoelectronic devices of two-dimensional materials, deterministic transfer for thin single-walled carbon nanotube (SWNT) film has not been realized. Herein, we report a transfer method of few hundreds nanometer-thick SWNT film to a desired prepatterned substrate position with few micrometer precision. This method comprises poly(methyl methacrylate) (PMMA)-coated SWNT film, creating surface anisotropy on both sides for SWNT film transfer, and two sets of translators manipulating the film to desired position and angle. Especially, adsorption and desorption of PMMA-coated SWNT film by each translator were modulated by use of interfacial water which not only promotes PMMA side to the transient sample loader via water capillary and releases the final substrate with capillary assisted sliding. Successful transfer of thin SWNT film with few tens of micrometer precision was achieved and will be beneficial for the preparation of high-end optoelectronic devices.

KEYWORDS
carbon nanotube, deterministic transfer, PMMA, thin film

Single-walled carbon nanotube (SWNT), hollow rolled-up of graphene sheet, possesses flexible, yet excellent electrical[1] and mechanical[2] properties, and has been utilized for various applications including thin film transistor[1] and thermoelectric devices.[3] Solution-processed SWNT dispersion and subsequent device fabrication require reliable methodologies including film formation and transfer for massive production.

SWNT film, typically random network structure of one-dimensional (1D) SWNT, can be prepared by various methods. Bulk filtration of SWNT dispersion on a paper filter can produce a micrometer-thick freestanding SWNT film. However, high-end SWNT applications such as flexible thin film transistor or thermoelectric device often require few tens of nanometer-thick SWNT film with specific size to a desired position. Among methods to prepare thin SWNT film, filtration method using a dissolvable membrane filter[4] has several advantages since thickness of SWNT film can be controlled by amount of dispersion to few tens of nanometer thickness, and selective solubility of filtration membrane toward various solvents gives a handle to prepare SWNT film onto a desired substrate by removing polymeric membrane.[4]

On the other hand, although deterministic transfer of 2D materials including graphene and transition metal dichalcogenides has been implemented for preparation of optoelectronic device,[5] such method including thin SWNT film has not been realized. Transfer method of such low dimensional materials to a desired substrate utilizes poly(methyl methacrylate) (PMMA) as a protective and sacrificial support[6] during transfer process. Although transfer method of those bulk films has been developed, a small piece of SWNT film in a deterministic way has not been achieved yet.

In this communication below, we developed a deterministic thin SWNT film transfer method for a given target substrate. For this, SWNT dispersion was prepared by a sonochemical method using *N*-dodecyl isoalloxazine (FC12) as a surfactant chemical. Thin SWNT film was prepared by successive filtration for SWNT film preparation, and PMMA coating. Importantly, by using x–y–z and x–y–θ translators, PMMA-coated SWNT film was transferred to a desired prepatterned substrate by using water as adhesion controller from an intermediate sample loader to a target substrate.

SWNT film was prepared by a filtration of SWNT dispersion. For the preparation SWNT dispersion, plasma torched SWNT (diameter $= 1.3 \pm 0.3$ nm, RN-220 SWNTs, batch# R26-036, NanoIntegris) was dispersed by using a sonochemical method (VCX 750, 40% power, 18.8 W/ml, probe tip diameter: 13 mm; Sonics & Materials) including FC12 as a surfactant, in *p*-xylene according to the previous

literature (see Experimental in Supporting Information for detailed method).[7–9] After centrifugation at 5000*g* where *g* is gravitational acceleration, the 80% supernatant was collected. The dispersion exhibits greenish dark color, indicating successful dispersion of SWNT, as shown in the photograph of Figure 1a. The absorption spectrum of this sample (Figure 1b) displays this sample was enriched semiconducting SWNT according to the literature.[8] Figure 1c–e illustrate stepwise procedure to make a PMMA-coated SWNT film. SWNT film was prepared by a filtration of the SWNT dispersion by using a dissolvable membrane filter consisting of nitrocellulose polymer (1 in. diameter; Advantec). After washing with copious amount of *p*-xylene, the SWNT film on the membrane was transferred to a quartz substrate (2.5 cm × 2.5 cm × 1 mm). The resulting substrate was located into a custom-made acetone reflux glassware to gently remove polymeric membrane by acetone vapor. During dissolution of membrane filter, white polymeric residues are spilled from the SWNT film.

After 1 day of exposure to acetone vapor, the dried SWNT film on a quartz was spincoated with a protective PMMA (molecular weight: 950 kDa; MicroChem) by using a spincoater with 1000 rpm for 1 min. This process produces 112 nm thick PMMA coating.[10] Freestanding SWNT film with top PMMA coat was obtained by treatment with buffered oxide etchant (BOE; 10:1 volume ratio, 36% of NH_4F and 4.7% of HF, etching rate: 1 nm/s at 25°C, Merck) of underlying quartz substrate for 2 h. Prior to BOE etching, one can obtain a piece of SWNT film by cutting the film using a razor blade.

Prior to transfer, we have set up a deterministic transfer stage as shown in Figure 2a. This stage contains one *x*–*y*–*z* translator for a glass sample loader manipulation, and another *x*–*y*–θ translator for target substrate manipulation (Figure 2b), along with ×0.75–4.5 magnification optics and a CMOS camera (Eakins) for observation. Overall setup was constructed with affordable cost.[11]

Water-assisted transfer steps of thin SWNT film were depicted in Figure 3a–c. For Step 1 of a deterministic transfer, PMMA-coated SWNT film was floated on a deionized (DI) water. The floating film was picked up by a DI water droplet hanging on hydrophilic glass sample loader. Importantly, water droplet forms water capillary to minimize the interaction between PMMA and glass sample loader otherwise PMMA-coated SWNT film permanently sticks to glass sample loader with deformation (Figure S2) due to strong surface interaction which will be discussed. In addition, water capillary is necessary to release the film on a given target.

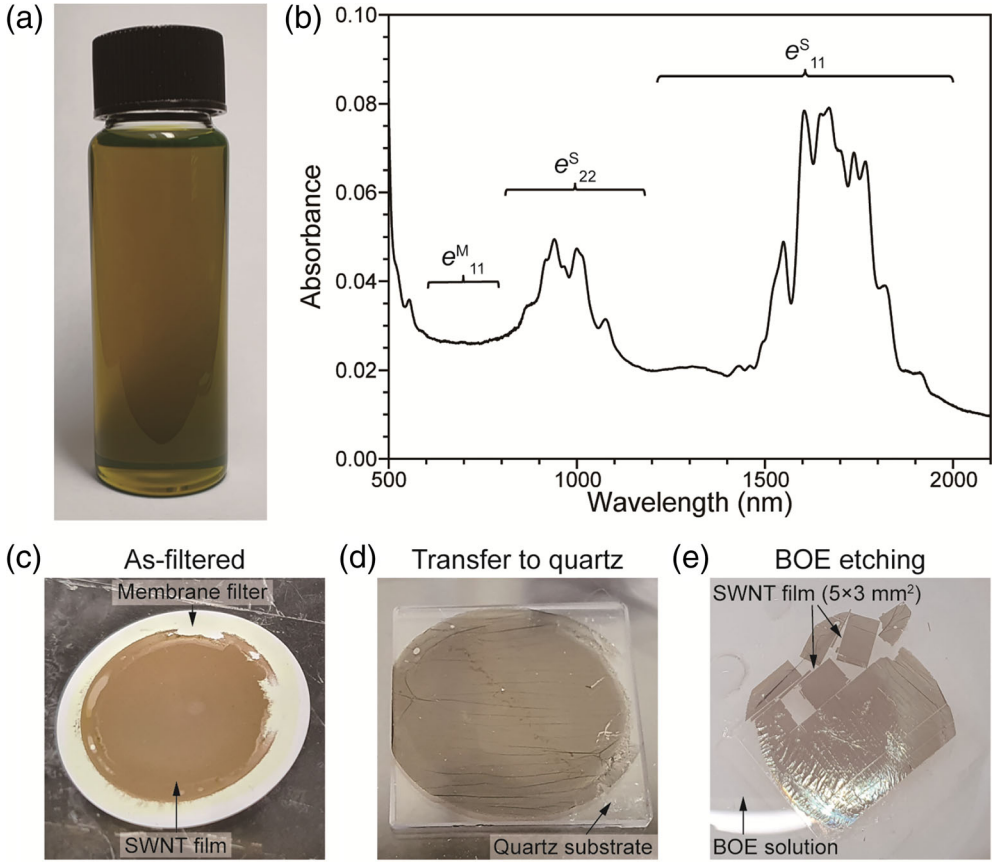

FIGURE 1 Preparation of PMMA-coated SWNT film: (a) A photograph and (b) UV–vis–NIR absorption spectrum of as-prepared FC12-PSWNT dispersion. Film preparation process: (c) Filtration of SWNT dispersion on a dissolvable membrane, (d) transfer of the film to a quartz substrate, and (e) floating PMMA-coated SWNT film by BOE etching. BOE, buffered oxide etchant; PMMA, poly(methyl methacrylate); SWNT, single-walled carbon nanotube

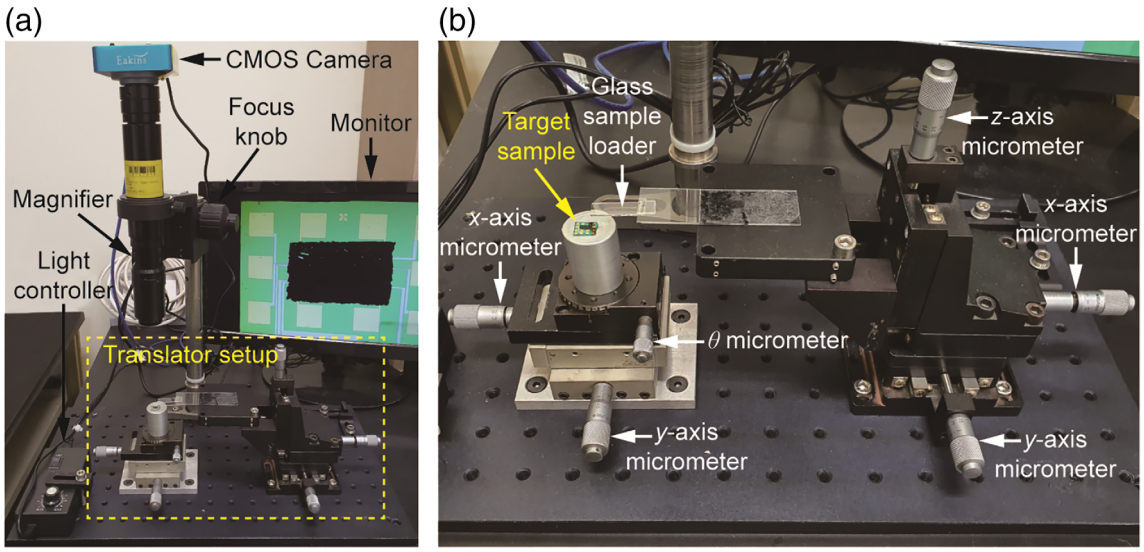

FIGURE 2 Photographs of (a) whole deterministic transfer setup including CMOS camera, monitor, and two translator sets, and (b) two translator sets manipulating *x*–*y*–*θ* of target substrate (left) and *x*–*y*–*z* of glass sample loader (right)

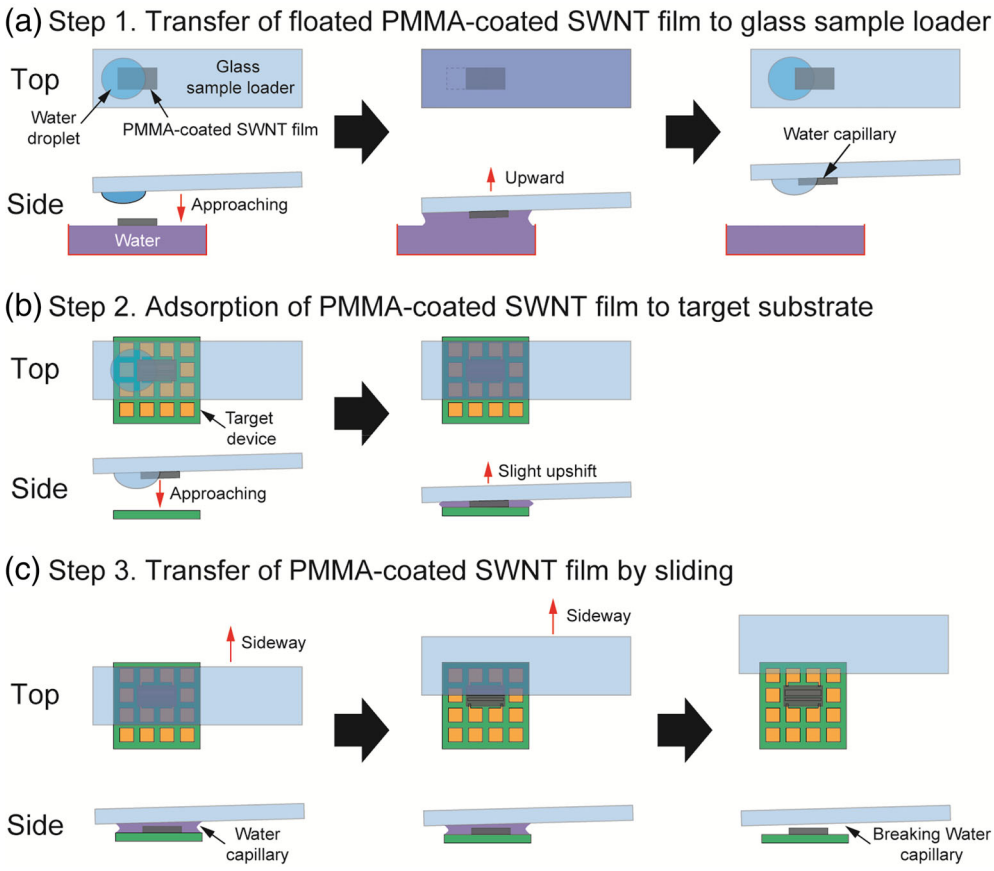

FIGURE 3 Procedure for a deterministic transfer of SWNT film. (a) Step 1: Transfer of floating PMMA-coated SWNT film to a glass sample loader. Note that PMMA coating of SWNT film is top side. (b) Step 2: Adsorption of SWNT film on glass sample loader to target substrate. (c) Step 3: Sliding-assisted transfer of SWNT film to a target substrate. PMMA, poly(methyl methacrylate); SWNT, single-walled carbon nanotube

In Step 2, the SWNT film on a glass sample loader was transferred to a target prepatterned device. This process involves approaching the SWNT film to a target substrate by manipulating two translator sets including sample rotation. Upon contact with underlying target substrate, SWNT film is immediately sticked on the target substrate

with maintained water capillary between PMMA and sample loader. The glass sample loader was slightly elevated with the maintained water capillary so that the interaction between PMMA and loader is slightly decoupled as illustrated in second scheme of Step 2. In Step 3, while maintaining water capillary, the glass sample loader was slowly moving parallel, leaving SWNT film behind in a target position. Note that parallel movement of the sample loader is necessary to gradually minimize PMMA-loader interaction controlled by water capillary. In addition, whole process needs to finish before water droplet dried.

Figure 4a–c display the corresponding photographs of each stage with a target substrate with 50 nm thick Au electrodes. Our goal is to transfer a 5×3 mm^2 PMMA-coated SWNT films with varying thickness to a desired position as shown in Figure S1. Figure 4a displays photographs before and after pickup of thin SWNT film by water droplet (5.9 ± 0.6 μl at 23°C) containing glass sample loader. It is noteworthy that amount of water was adjusted to form water capillary according to the size of SWNT film. Excess water is desirable to prevent drying-induced permanent stick of PMMA-coated SWNT film. After the pickup process, water capillary was formed in between PMMA side of thin SWNT film and glass sample loader and the role of water will be elaborated in later discussion. Figure 4b display contact-initiated adsorption of SWNT film to a target substrate. Prior to adsorption, it was possible to fine align SWNT film to target substrate. Final Step 3 comprises sliding motion of slightly elevated glass sample loader to sideway. During this process, water capillary between PMMA coating and glass sample loader leaves the film as adsorbed. During this motion, water capillary is maintained between glass sample loader and target substrate, as evident by top photograph of Figure 4c. Moreover, the water capillary at the interface acts as a lubricant, prevents the SWNT film from dragging or tearing in the process of sliding the sample loader, and allows the SWNT film to be positioned at the desired position (Figure S3). Importantly, nearly equal space of SWNT film between Au electrodes was achieved by this deterministic transfer method. As shown in Figures 4 and S1, SWNT films were precisely attached to the halfway of four thin electrodes from the large Au pad, showing few tens micrometer precision of transfer. After drying water, thin SWNT film on a target substrate was treated with acetone to remove PMMA. Although a series of transfer step utilizes thick SWNT film for visuality, it is possible to have thin SWNT film as thin as 165 nm confirmed by AFM height topography (Figure S5A,B for AFM topography of thin SWNT film and their height profile).

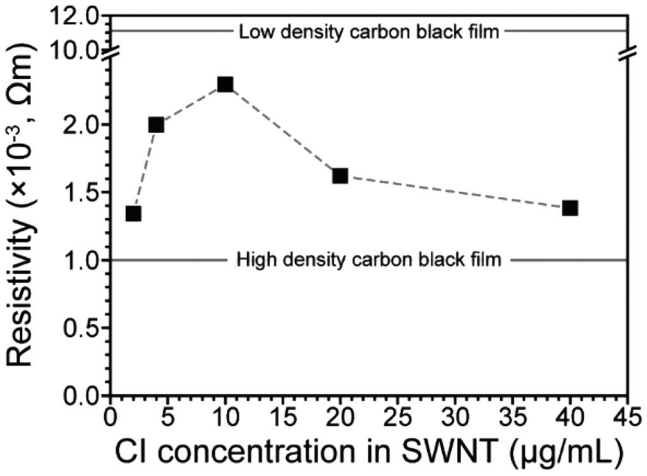

FIGURE 5 Resistivity trend of PSWNT film with different loading amounts of Cl. Resistivity of low[15] and high density[16] carbon black film is drawn in solid line

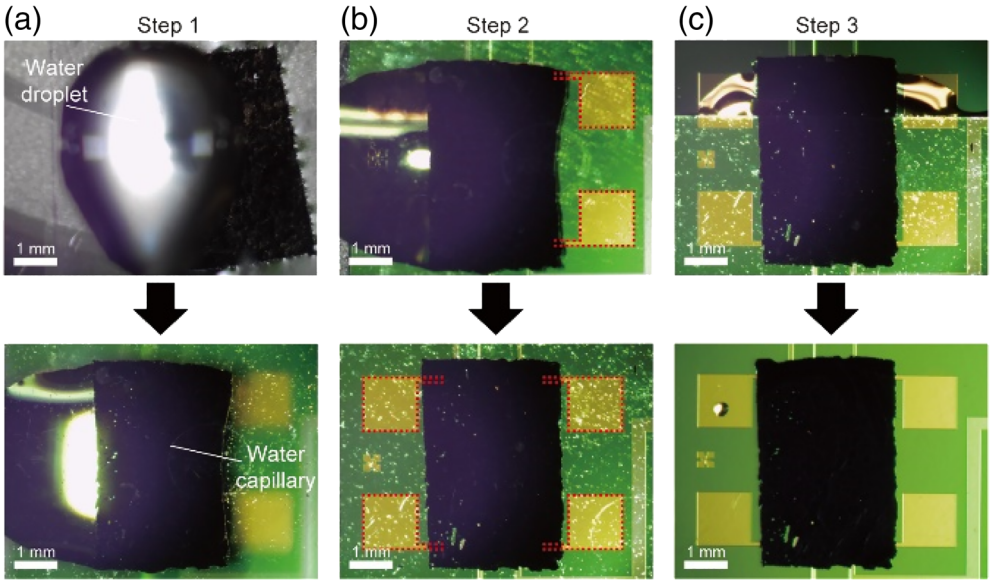

FIGURE 4 Photographs of each transfer step. The thin electrodes (width: 100 μm, length: 560 μm) from the large square Au pad are drawn as dotted line

TABLE 1 Comparison of contact angle of various surfaces

	SWNT film	PMMA	Bare SiO_2	SiO_2/Si wafer
Contact angle (°)	86.1 ± 19.0	71.4 ± 1.2	4.4 ± 0.4	77.7 ± 3.8

Abrreviations: PMMA, poly(methyl methacrylate); SWNT, single-walled carbon nanotube.

Next, the electrical contact between SWNT film and electrodes was addressed. Resistivity of SWNT films were measured by two probe method.[12] For this, carbonaceous impurity (CI) which is graphitic nanoparticle was intentionally added to modulate electrical resistivity. Figure 5 shows resistivity trend with increasing CI contents in SWNT dispersion. The thin film prepared by as-dispersed SWNT displays resistivity of 1.35 mΩ·m. Increasing CI displays initial increased resistivity and later restored resistivity. This resistivity behavior can be explained by electrical percolation theory of SWNT and CI used as conduction channel. Initial increase in resistivity of the film in presence of small amount of CI can be explained by scattering of charge carrier along SWNT network[13] due to CI which is known to have good interaction with SWNT sidewalls.[14] When CI concentration exceeds percolation threshold by itself, resistivity becomes smaller. In our case, the percolation threshold of CI can be estimated to be around 10 μg/ml of the CI concentration. Although clear mechanism for electrical conductivity in the presence of predominantly CI and small amounts of SWNTs was not elucidated, SWNT film provides good contact with underlying prepatterned substrate.

The remaining question to be addressed is role of water for transfer of PMMA-coated SWNT film. As shown in Table 1 and Figure S6 in the SI, it is well-known that SWNT film is hydrophobic, showing contact angle of water to be 86° while contact angle of PMMA is less hydrophobic (i.e., 71°). Those contrasts in contact angle were implemented to manipulate film transfer. Since bare SiO_2 is hydrophilic (4.4°, varied by condition), it exhibits strong interaction with PMMA. Decoupling of this interaction was possible by addition of water capillary. In addition, hydrophobic SWNT film is prone to adhere to patterned substrate having similar contact angle (78°), rather than interfacing with water during Step 2. In this process, the aforementioned water capillary helps to detach the SWNT film easily from the sample loader to achieve successful transfer, and furthermore, minimizes the dislocation of the SWNT film from the desired position when sliding and removing the sample loader with gradually receding of waterfront.

In summary, we developed a transfer method of thin SWNT film to a desired substrate. Janus-like surface energies of PMMA-coated SWNT film along with interfacial water enable to modulate substrate-to-substrate transfer of SWNT film. Two translator sets help to approach the sample to desired position with few tens of micrometer precision. SWNT film connected to electrode shows good electrical contact with underlying electrodes. This method will be beneficial to transfer thin SWNT film with specific size and thickness to a desired substrate, and ultimately leads to high-performance SWNT-based thin film transistor and thermoelectric devices.

ACKNOWLEDGMENTS

The authors acknowledge the invitation for special issue of Physical Chemistry Division of Korean Chemical Society in this Journal. This research was mainly supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science, and Technology (2020R1F1A1076983 and 2020R1A4A1017737).

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SUPPORTING INFORMATION

Additional supporting information may be found in the online version of the article at the publisher's website.

How to cite this article: M. Park, S.-Y. Ju, *Bull. Korean Chem. Soc* **2022**, *43*(2), 196. https://doi.org/10.1002/bkcs.12462

Deterministic Transfer of Thin Carbon Nanotube Film

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Author Contributions

S. J. conceived the idea and wrote the manuscript. M. P. performed transferring films, formal analysis, and supporting writing. All figures were drawn by authors.

((Please specify the contributions of each author including the type (e.g. data curation, funding acquisition, formal analysis, investigation, project administration, validation, writing of original draft) and the degree (e.g. lead, equal, supporting) of contribution.))

Table of Contents

Experimental

Materials and Instrumentation: *p*-xylene and acetone were purchased from Alfa Aesar(MA, USA) and Samchun chemical (Republic of Korea), respectively. All solvents and chemicals were reagent grade and were used as received. As-produced type termed as PSWNT (RN-220 SWNTs, batch# R26-036, nanotube purity of 30-70%, NanoIntegris, Canada) is used. Diameter (d_t) distribution of PSWNT is 1.3 ± 0.4 nm. All the measurements were carried out at room temperature unless otherwise noted. Slide glass which used in this experiment was consist of hydrophilic soda lime glass (#1000412, Marienfield-Superior, Germany).

Absorption spectrum measurement: UV-vis-NIR absorption spectra up to 2100 nm were recorded on a JASCO V-770 (Japan) with absorption cuvettes having 1 mm path length (21/Q/1, Starna scientific, UK). Absorbances were measured *via* double beam configuration. UV-vis regions are dispersed by 1200 lines/mm grating and NIR region is dispersed by 300 lines/mm grating. D_2 and halogen lamps were used for excitations for 190 to 350 nm and 330 nm to 3200 nm, respectively. Wavelength accuracy was 0.3 nm for UV-vis and 1.5 nm for NIR regions, respectively. Photometric accuracy is 0.0015 for 0 to 0.5 abs, and 0.0025 abs for 0.5 to 1 abs. Detectors for UV-vis and NIR regions were photomultiplier tube and PbS photoconductive cell.

Atomic force microscope (AFM) measurement: AFM measurement was conducted by using a commercially available AFM (NX10, Park systems, Republic of Korea). An Al-coated silicon cantilever with a spring constant 37 N/m, a resonance frequency of 300 kHz, and quoted radius of ca. 6 nm (ACTA, App Nano, CA, USA) was utilized to measure height topographies. Typically, a 512 × 512 pixel image was collected from 5 μm × 5 μm size area. The measured height topographies were analyzed by XEI 4.3.4 program (Park systems, Republic of Korea).

SWNT dispersions: a mixture of 1 mg of as-prepared PSWNT and 1 mg of *N*-dodecyl isoalloxazine was added to 4 mL of *p*-xylene which was dried over 3Å molecular sieve (Alfa Aesar, MA, USA) overnight prior to use. The resulting mixture was subjected to brief bath sonication (5 min, Branson 1510, 70 W, Emerson, MO, USA) for mixing, and further to 1 h tip sonication [40% power, 18.8 W/mL, probe tip diameter: 13 mm, VCX 750, Sonics & Materials, CT, USA]. During the sonication, the temperature of the sample vials was maintained at 15 °C by the external water circulator (Lab Companion RW-2025G, Jeio Tech, Republic of Korea). The centrifugations with different *g* forces (5 and 30 k*g*) were conducted by high performance centrifuge with a fixed angle rotor (Avanti J-26 XPI and JA-25.50, respectively, Beckman Coulter, IN, USA) at the room temperature using organic solvent-tolerant centrifugal tube (50 mL, Cat. #: 3114-0050, fluorinated poly(ethylene) (PE)-co-poly(propylene) (PP), Nalgene, NY, USA). The 80% supernatant was carefully collected for the further measurements.

SWNT film preparation: The acetone-dissolvable membrane filter was used to prepare PSWNT film on a quartz substrate. 0.1 μm pore-sized mixed cellulose ester (MCE) membrane filter (1" in diameter, cat. #: A010A025A, Advantec, Tokyo, Japan) was used to filter *ca*. 3 mL of PSWNT dispersion for further treatment. To prepare SWNT film, the membrane filter was placed in upside down manner on a quartz substrate (2.5 cm × 2.5 cm × 1 mm, NC-200, Hanjin Quartz, Republic of Korea) having 20 μL of deionized (DI) water droplet to promote better adhesion. A dummy top slide glass was placed and pressurized on top of the membrane filter/SWNT film for few sec to promote evenly adhered membrane filter on SWNT. After dummy slide glass was removed, the entrapped DI water was carefully removed by a tissue wiper along the perimeter of the membrane filter, and the membrane-adhered quartz substrate was quickly loaded into a custom-made acetone vapor reflux chamber prior to dryness. Special care was taken by placing the quartz substrate not to direct

contact acetone drop condensed by a reflux condenser. The dissolution of membrane filter can be observed by melting out a whitish membrane residue within few h and the complete removal required two days.

Deterministic transfer of SWNT film to a prepatterned device: The SWNT film transfer process was adopted from poly(methyl methacrylate) (PMMA)-assisted graphene transfer method.[S1-S2] Deterministic transfer of thin SWNT film follows the protocol. In order to transfer the SWNT film on quartz substrate to a prepatterned chip, PSWNT film-placed quartz substrate was initially spincoated by PMMA (molecular weight: 950 kDa, 2% dilution in anisole (A2), viscosity: 10.8 cP, density: 0.996 g/mL, MicroChem, MA, USA) which is standard materials to fabricate known thickness with various rpm.[S3] The spincoating process was conducted at 1000 rpm for 60 sec (SF-1A, BGK, Republic of Korea) to form 112 nm thick PMMA layer on SWNT film. After that, PMMA-coated SWNT film was cut carefully by a sharp blade to make the rectangular film (5 mm × 3 mm size) which fits to the desired area of pre-patterned chip. The PMMA-coated SWNT film was carefully detached from quartz substrate by floating them on a buffered oxide etchant (10:1 volume ratio, 36% of NH_4F and 4.7% of HF, etching rate: 1 nm/sec at 25 °C, Merck, Germany) for 2 h. The floated PSWNT film with the PMMA coating was scooped out by a clean slide glass and washed the residual oxide etchant subsequently with DI water two times. The rectangular SWNT film was transferred to exact position of pre-patterned chip (lithographically defined Pt electrodes (100 nm) on 300 μm thick (100) Si substrate *via* custom-made transfer system with *x*-*y*-*z* and *x*-*y*-*θ* translators which is controlled by using micrometers (minimum tick interval: 10 μm, Mitutoyo, Japan). To remove the PMMA coating on PSWNT film, the chip was immersed in clean acetone bath overnight and dried at 90 °C under 10^{-3} torr vacuum. The intimate contact between PSWNT film and Au electrodes was confirmed by two probe measurement.

Supporting figures

Figure S1. (A-E) PMMA-coated SWNT film to a desired position.

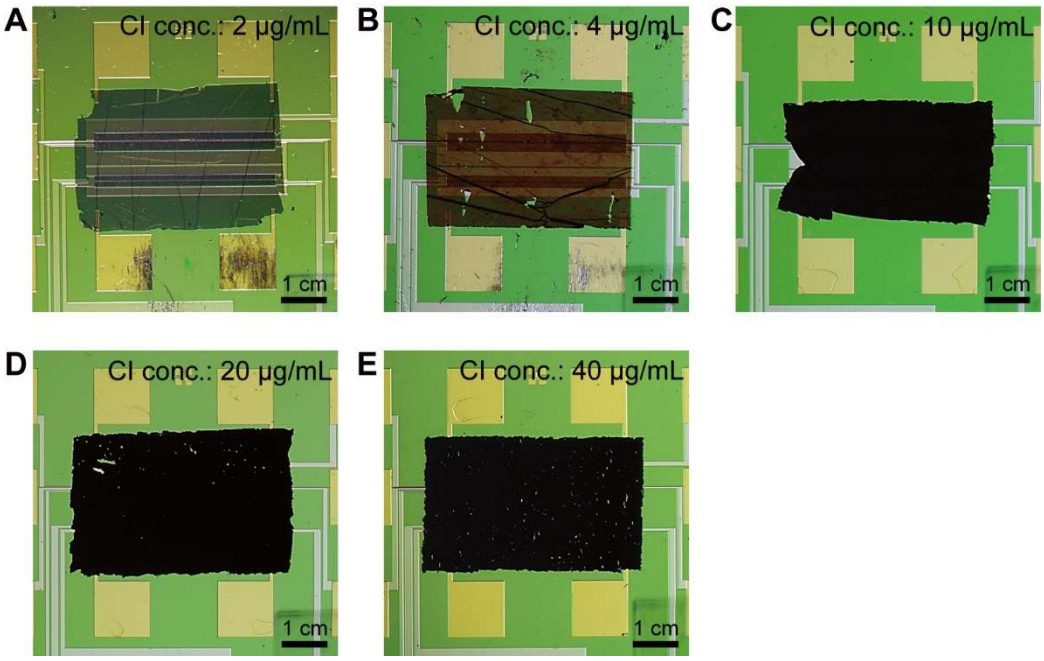


Figure S2. Deformed SWNT film on pickup by sample loader in the absence of initial water droplet.

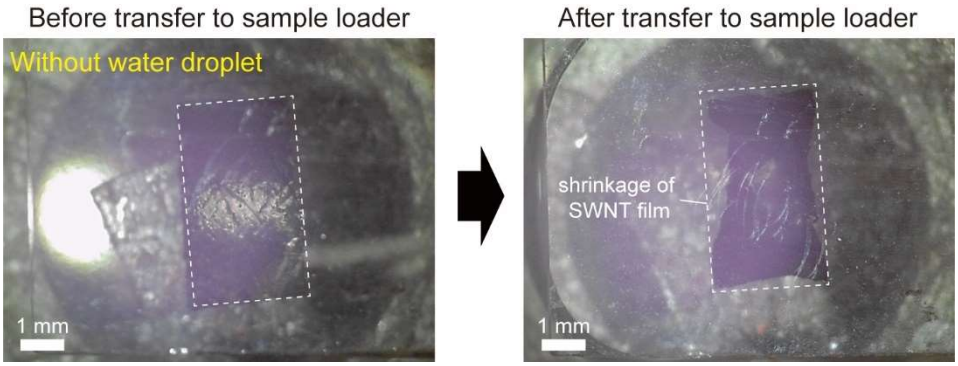


Figure S3. Stable adsorption of SWNT film onto the substrate during the sample loader sliding.

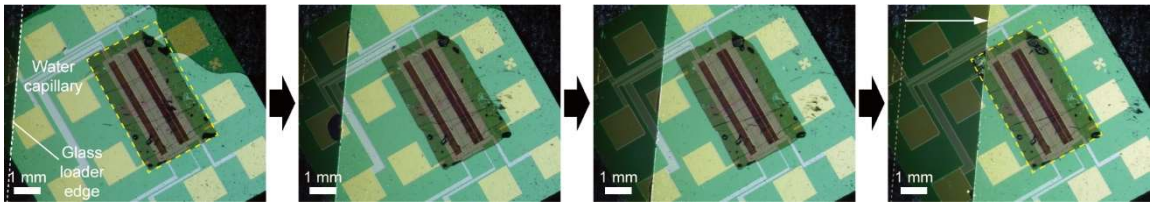


Figure S4. (A) AFM height topography of PSWNT thin film and (B) its height profile from red triangle region. Dotted line denotes the average height of thin SWNT film.

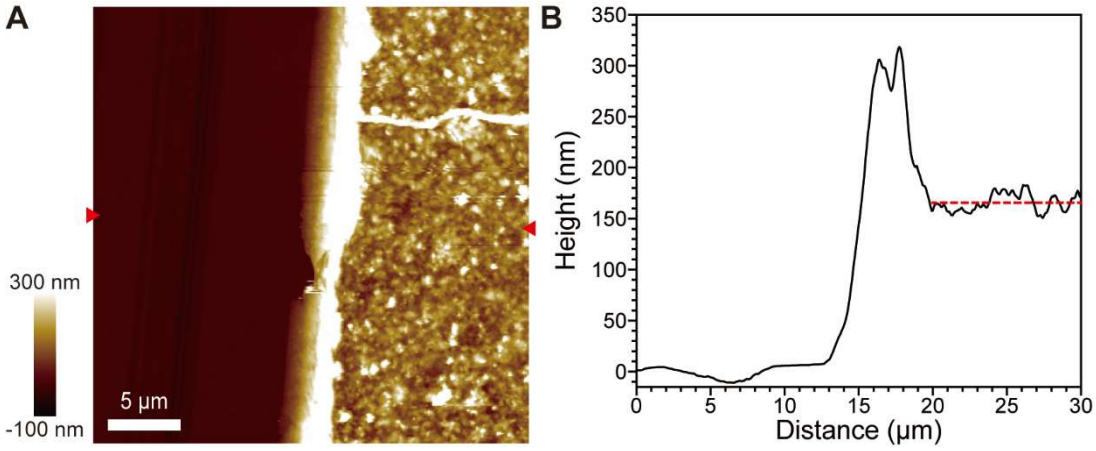
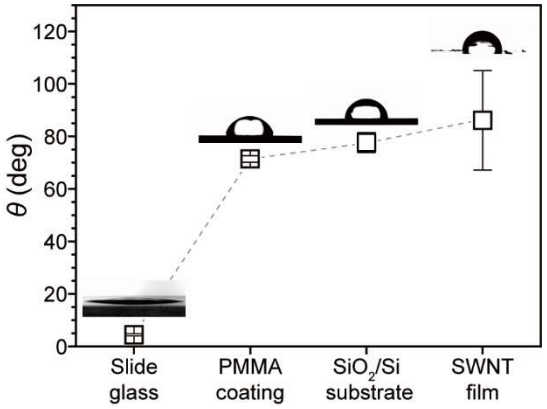


Figure S5. Contact angle measurement of slide glass, PMMA coating, SiO_2/Si substrate, and SWNT film.



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