Nanolithographic patterning of transparent, conductive single-walled carbon nanotube films by inductively coupled plasma reactive ion etching

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The authors report successful patterning of transparent, conductive single-walled carbon nanotube films down to 100 nm lateral dimensions by photolithography or e-beam lithography and subsequent O₂ plasma etching using an inductively coupled plasma reactive ion etching (ICP-RIE) system. They systematically study the effect of ICP-RIE etch parameters, such as substrate bias power, chamber pressure, and substrate cooling, on the nanotube film etch rate and etch selectivity. They also characterize the effect of the linewidth etched on the nanotube film etch rate for widths ranging from 50 μm down to 100 nm. Furthermore, by fabricating standard four point probe structures using the patterning capability developed, the authors investigate the effect of different resist processes on the resistivity of patterned single-walled carbon nanotube films and the effect of ICP reactive ion etching on the resistivity of partially etched nanotube films. In addition, they demonstrate that using an ICP-RIE system provides significant advantages, such as faster etch rates and better etch selectivity, over conventional parallel plate RIE plasma systems, making it possible to pattern lateral features as small as 100 nm in nanotube films. The simple and efficient “top-down” patterning capability developed in this article could open up many opportunities for integrating single-walled nanotube films into a wide range of electronic and optoelectronic devices. © 2007 American Vacuum Society. [DOI: 10.1116/1.2699836]

I. INTRODUCTION

Single-walled carbon nanotubes (SWNTs) have attracted significant research attention in the last decade because of their remarkable physical and electronic properties, such as high mobility and current density.¹ Many discrete electronic devices based on individual SWCNTs, such as transistors and sensors, have been experimentally demonstrated.²³ Despite these outstanding properties, however, controlling the diameter, chirality, location, and direction of individual nanotubes has proven very challenging.⁴

A single-walled nanotube film (SWNT film) is a threedimensional film of tens of nanometers thickness, consisting of an interwoven mesh of single-walled nanotubes. For SWNT films, individual variations in diameter and chirality are ensemble averaged to yield uniform physical and electronic properties.⁵⁻⁸ As a result, the reproducibility and reliability problems found in individual nanotubes are solved, and carbon nanotube film based devices can easily be mass produced in a cost effective manner. Furthermore, what makes SWNT films even more attractive for device applications is that they are flexible, transparent, and conductive. SWNT films have a resistivity on the order of 10⁻⁴ Ω cm, and they are also optically transparent over the visible and near-infrared portions of the spectrum. These outstanding properties have established SWNT films as a new class of optically transparent and electrically conducting materials that can be used in applications such as thin film transistors,⁹,¹⁰ flexible microelectronics¹¹⁻¹³ chemical sensors,¹⁴⁻¹⁷ and optoelectronic devices.¹⁸⁻²¹

Any potential device application utilizing SWNT films requires the capability to efficiently pattern them. Nanotube networks (which are dilute two-dimensional interconnected networks of nanotubes forming a submonolayer over a substrate) have been patterned recently by a variety of techniques including the use of a CO₂ snow jet,⁶ transfer printing,⁷,²² and O₂ plasma etching⁴ in the micron regime. In addition, more recently, nanotube films with various thicknesses have been patterned into micron size features using photolithography and inductively coupled plasma (ICP) reactive ion etching (RIE).²³ Although linewidths down to about 1.5 μm have been successfully patterned using this method, patterning of submicron features in SWNT films has

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not been demonstrated previously. Moreover, the effect of ICP-RIE etch parameters on the SWNT film etch rate and etch selectivity has not been systematically characterized. In this article, we use photolithography or e-beam lithography, and subsequent O2 plasma etching in an ICP-RIE system to pattern nanotube films down to submicron lateral dimensions. We experimentally show that features with linewidths less than 100 nm can be successfully patterned using this technique with good selectivity and directionality. In addition, we systematically study the effect of ICP-RIE etch parameters, such as the substrate bias power and chamber pressure, on the SWNT film etch rate and etch selectivity. We also compare O2 plasma etching of SWNT films in an ICP-RIE system to that in a conventional parallel plate RIE system. We find that using an ICP-RIE system significantly increases the SWNT film etch rate and improves the etch selectivity between the SWNT film and polymethylmethacrylate (PMMA) compared to a conventional RIE system, making it possible to pattern SWNT films down to ~100 nm lateral dimensions by e-beam lithography. Furthermore, by fabricating standard four point probe structures using the patterning capability developed, we investigate the effect of different resist processes on the resistivity of patterned SWNT films. The “top-down” patterning capability reported in this article could open up many opportunities for fabricating SWNT film based electronic and optoelectronic devices.

II. EXPERIMENTAL PROCEDURE

SWNT films were deposited by a vacuum filtration method as described in detail previously. In summary, a dilute suspension of purified SWNTs was vacuum filtered onto a filtration membrane. The nanotubes deposit as a thin film on the membrane with the thickness of the film controlled by the concentration of nanotubes in the suspension and the volume of the suspension filtered. The film can then be transferred onto a desired substrate by placing the film side against the substrate, applying pressure, and drying the film. To complete the process, the filtration membrane is dissolved in a solvent, leaving only the nanotube film adhered to the substrate. The substrates used in this work were (100) silicon with a 500 nm layer of thermally grown SiO2 on top.

Following the deposition step, the SWNT film was patterned either by photolithography or e-beam lithography. For photolithography, three different types of resist processes were used as the mask. The first process used a 1.3 μm thick layer of Shipley Microposit S1813 photoresist. It was found by extensive atomic force microscopy (AFM) imaging that when the S1813 resist is deposited directly on top of individual nanotubes, it leaves a residue. On the other hand, it was observed that Microchem LOR3B lift-off resist and PMMA do not contaminate the nanotubes. The LOR3B lift-off resist can be removed by standard developer solutions, with the dissolution rate determined primarily by the prebake temperature. To protect the SWNT film from potential contamination due to the S1813 resist, for the second process, a dual layer resist structure consisting of a 1.3 μm thick S1813 layer on top of a 250 nm thick LOR3B layer was used. Finally, for the third process, a dual layer resist process consisting of a 1.3 μm thick Shipley S1813 layer on top of a 250 nm thick PMMA layer (950 K, 4% in anisole) was used. Since the S1813 resist is not in direct contact with the nanotubes in the second and third processes, no residue is left on the nanotube film during fabrication. However, PMMA cannot be exposed by the 365 nm light source available in the Karl Suss MA-6 contact mask aligner that was used for photolithography. As a result, for the third process, the S1813 layer was first exposed by the mask aligner and developed. Subsequently, the PMMA layer was patterned by O2 plasma etching with the S1813 layer acting as the mask. In all three resist processes, Shipley Microposit MF319 was used as the developer. The particular resist process used was found to affect the resistivity of the SWNT film, as presented in detail later.

For e-beam lithography, a single layer of PMMA (950 K, 2% or 4% in anisole depending on the feature size patterned and PMMA thickness desired) was used as the masking layer, and a Raith 150 e-beam writer was used for exposure. After exposure and development, the nanotube film not protected by the resist mask was etched using an O2 chemistry in a Unaxis Shuttlelock ICP-RIE system. The schematic of the ICP etcher is shown in Fig. 1. The ICP-RIE system decouples plasma density (controlled by the ICP power supply) and ion energy (controlled by the substrate power supply). As a result, compared to conventional diode RIE systems, very high plasma densities (>1011 ions cm−3) can be achieved at lower pressures, resulting in more anisotropic etch profiles and significantly higher etch rates. Etching in ICP systems has a large physical component combined with a smaller chemical component. O2 plasma is commonly used for removing organic materials such as photoresist and has also been used to etch carbon nanotubes. The reaction between oxygen and organic materials produces volatile species such as CO and CO2, which are pumped out during the etch process. The etch parameters for our initial SWNT etch recipe were 300 W power on the 2 MHz ICP rf supply, 100 W power on the 13.56 MHz substrate rf supply, 45 mTorr chamber pressure, and a 20 sccm O2 flow rate. In addition, a helium flow rate of 10 sccm was used to cool...
III. RESULTS AND DISCUSSION

Resulting SWNT film etch profiles were characterized by a Digital Instruments Nanoscope III AFM. Figure 2(a) shows the AFM image of a ~3 μm line etched in a ~20 nm thick nanotube film using the LOR3B/S1813 dual resist photolithography process and the initial ICP etch recipe given previously. The cross-sectional height profile for the same AFM image is plotted in Fig. 2(b), showing clearly the transition between the film and the etched regions. Similar etch profiles were obtained using the other two resist processes described previously. Figure 2(c) shows an AFM image of a series of lines with nanotube film width and spacing of about 200 nm patterned by e-beam lithography and ICP etching of a SWNT film of about 14 nm thickness. The cross-sectional height profile for the same AFM image is plotted in Fig. 2(d), showing a clear transition between the film and the etched regions even at these submicron lateral dimensions. The etch profiles in Figs. 2(b) and 2(d) are not sufficient to determine how sharp the actual etch profile is since the radius of curvature and the sidewall angle of the AFM tip could decrease the sharpness of the observed transition profile. Furthermore, Figs. 3(a) and 3(b) show AFM and scanning electron microscopy (SEM) images, respectively, of letters printed in nanotube film using e-beam lithography and ICP etching. The width of the text characters are on the order of 200–300 nm and the SWNT film thickness is 20 nm. The scale bars are 1 and 2 μm in parts (a) and (b), respectively.

In order to characterize quantitatively the SWNT film and resist etch rates using the initial ICP-RIE etch recipe given previously, a series of lines with equal width and spacing were partially etched in 50–100 nm thick nanotube films, such that some nanotube films still remained in the etched areas. Figure 4(a) shows an AFM image of such a series of lines with ~200 nm width and spacing partially etched in a 75 nm thick SWNT film. Unlike the lines in Fig. 2(c), the lines in Fig. 4(a) have not been etched all the way down to the substrate, as evident from the texture of the remaining film visible in the etched areas. By measuring the height difference between the partially etched and non-etched film lines using cross-sectional AFM analysis, the average etch depth, and as a result, the etch rate can be calculated. For example, Fig. 4(b) shows the cross-sectional AFM profile for the lines shown in Fig. 4(a), giving an average etch depth of about 19 nm for this particular sample. Dividing this depth by the etch time of 8 s, a nanotube film etch rate of ~2.4 nm/s is obtained. The S1813, LOR3B, and PMMA etch rates were determined by measuring the initial and final

![Image](image-url)
resist thicknesses using a Nanometrics Nanospec spectrometer and dividing by the etch time. Using the initial recipe (i.e., 300 W ICP power, 100 W substrate bias power, 45 mTorr chamber pressure, 20 sccm O2 flow rate, and 10 sccm helium flow rate for substrate cooling), etch rates of 2.37, 4.59, 4.58, and 6.65 nm/s were observed for SWNT film, S1813, LOR3B, and PMMA, respectively, as listed in the first column of Table I. The error bar on these etch rates is approximately ±10%. The SWNT film etch rate is similar in magnitude to the ~4 nm/s observed in recent work using another ICP system. For SWNT films of tens of nanometer thickness, such as those used in this work, the 2.37 nm/s etch rate of the initial recipe provides both reasonably short etch times and a good control of the etch uniformity. The selectivity S of the etch between the nanotube film and the resist mask is defined by

$$S = \frac{r_{\text{SWNT}}}{r_{\text{resist}}}$$

(1)

where $r_{\text{SWNT}}$ is the etch rate of the SWNT film and $r_{\text{resist}}$ is the etch rate of the particular resist used as the mask. Using this definition, selectivity values of 1:1.94, 1:1.93, and 1:2.81 are obtained for S1813, LOR3B, and PMMA masking layers, respectively. Carbon nanotubes are much harder to etch compared to photoresists since they are chemically resistant and structurally stable. As a result, the etch rate of the SWNT film is slower than that of resists in an O2 plasma, and the selectivity values are less than unity. Since the resists are used as the etch mask, they need to be thick enough to withstand the nanotube film etch. The minimum resist thickness required for a given SWNT etch process is determined by the selectivity S of the etch process. Typical S1813 only and LOR3B/S1813 dual layer resist thicknesses used for photolithography are larger than 1 μm; as a result, based on the selectivity values given above, hundreds of nanometer thick SWNT films can easily be patterned by photolithography. More importantly, since the PMMA etch rate is not significantly higher than the nanotube film etch rate, typical PMMA thicknesses necessary for e-beam lithography (100–300 nm) can be used to pattern thin SWNT films (i.e., less than 100 nm) down to very small (<100 nm) lateral features. In short, although the etch selectivity between the SWNT film and PMMA is less than unity (S<0.36), it is still large enough to allow for e-beam patterning of SWNT films.

Aspect ratio dependent etching has been observed in some etch processes, such as silicon trench etching, resulting in a lower etch rate for smaller width trenches. Using the approach described in the preceding paragraph, we systematically studied the effect of the linewidth on the nanotube film etch rate for widths ranging from 50 μm all the way down to 100 nm. The spacing between the etched lines was set equal to the width of the lines in all cases. The etch rate was found to be almost constant at 2.37±0.3 nm/s, independent of the linewidth etched. This is most likely due to the fact that all our samples have a SWNT film thickness t<100 nm. The aspect ratio (AR) of the nanotube film etched, defined as $t/w$, where w is the width of the line etched, always satisfies $AR<1$ for all the samples. In other words, the plasma density is high enough and the aspect ratio is small enough so that reactant species are able to make it to the bottom of the etched lines even for the smallest (100 nm) linewidths.

Furthermore, we systematically studied the effect of changing various ICP etch parameters on the etch rates of the SWNT film and different resists, as listed in Table I, using the procedure described previously. To investigate the effect of the substrate bias power on the etch rate, we decreased the substrate power from 100 to 15 W, keeping all the other etch parameters constant as in the initial recipe. Table I shows that the nanotube film and resist etch rates are decreased by about a factor of 10 compared to those of the initial recipe. By reducing the substrate bias, the ion energy is reduced, resulting in a substantially slower etch rate. A slow etch rate could be useful in applications where the SWNT film thickness is very small and the etch rate and uniformity needs to be precisely controlled.

<table>
<thead>
<tr>
<th>ICP-RIE system etch rates (nm/s)</th>
<th>Parallel plate RIE system etch rate (nm/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Initial recipe</td>
</tr>
<tr>
<td>SWNT film</td>
<td>2.37</td>
</tr>
<tr>
<td>S1813</td>
<td>4.59</td>
</tr>
<tr>
<td>LOR3B</td>
<td>4.58</td>
</tr>
<tr>
<td>PMMA</td>
<td>6.65</td>
</tr>
</tbody>
</table>

FIG. 4. (a) AFM image of a series of nanotube film lines having equal widths (and spacings) of ~200 nm partially etched in a 75 nm thick SWNT film by e-beam lithography and ICP-RIE etching, as described in the text. Unlike the lines shown in the AFM image of Fig. 2(c), the lines in this AFM image have not been etched all the way down to the substrate, as evident from the texture of the remaining film mesh visible in the etched areas. The scale bar is 200 nm. (b) Cross-sectional height data for the AFM image of part (a), showing an average etch depth of about 19 nm for this particular sample. The etch rate can be calculated by dividing this etch depth by the total etch time.

TABLE I. Etch rates of the SWNT film and three different resists (S1813, LOR3B, and PMMA) under different plasma etch conditions using the Unaxis Shuttlelock ICP-RIE system and the Plasma Sciences RIE 200 W system. The initial ICP-RIE recipe in column I corresponds to an ICP power of 300 W, substrate bias power of 100 W, chamber pressure of 45 mTorr, and O2 flow rate of 20 sccm. In addition, a helium flow rate of 10 sccm was used to cool down the substrate. The headings of the other columns indicate the parameters that have changed compared to the initial recipe, with all the other parameters kept constant. The parallel plate RIE system etch parameters were rf power of 20 W, O2 flow rate of 12.5 sccm and chamber pressure of 140 mTorr.
To investigate the effect of chamber pressure on the etch rate, we decreased the chamber pressure from 45 to 10 mTorr, keeping all the other etch parameters constant as in the initial recipe. Table I shows that the nanotube film and resist etch rates increase by a factor between 1.7 and 3.5 compared to those of the initial recipe. A lower chamber pressure results in a more directed etch, higher ion energy, and increased etch rates due to fewer gas-phase collisions. A faster etch rate could be useful in applications where the SWNT film thickness is large. Furthermore, by taking the ratio of the etch rates listed in Table I, selectivity values of 1:0.95, 1:1.21, and 1:1.59 are obtained for S1813, LOR3B, and PMMA masking layers, respectively. These selectivity values are higher than those of the initial recipe. This is likely due to an increase in the physical etch component, which etches the nanotube film and resists at similar rates. In addition, increasing the chamber pressure from 45 to 100 mTorr (maximum pressure achievable in our system) was found not to change the etch rates of the nanotube film and resists significantly, showing that the etch rate has already saturated at 45 mTorr pressure.

Furthermore, we have investigated the effect of substrate cooling on the etch rates of the SWNT film and resists. Increasing the helium flow rate (which actively cools the substrate) from 10 to 40 sccm, keeping all other etch parameters constant as in the initial recipe, did not change the etch rates of the SWNT film and resists compared to those of the initial recipe. The optimum etch conditions depend on the nanotube film thickness that needs to be etched. Based on our results, for thick films, etching at low pressures would be the best option. On the other hand, for thin films, where the etch rate and uniformity needs to be better controlled, etching at low substrate bias power would be the best choice. For intermediate thicknesses, the initial recipe would work the best. Furthermore, our experiments indicate that the substrate bias power can be used to control the nanotube film etch rate without significantly changing the selectivity. Therefore, one can lower the chamber pressure to achieve the best selectivity and then adjust the substrate bias power to achieve the optimum etch rate based on the film thickness.

To compare the etch rates of the SWNT film and the three resists in an ICP-RIE system to those in a conventional parallel plate RIE system, we have also etched the SWNT film and resists using a Plasma Sciences RIE 200 W etcher, in which there is only one rf power source of 13.56 MHz frequency, and as a result, the plasma density and the ion energy are no longer decoupled. Using a rf power of 20 W, an O2 flow rate of 12.5 sccm, and a chamber pressure of 140 mTorr, we have observed that the etch rates of both the SWNT film and resists are substantially lower in this system, as listed in the last column of Table I. The etch rate of the SWNT film in the conventional RIE system was 0.05 nm/s, which is about five times slower than that in the ICP-RIE system even with a low substrate bias power of 15 W (See Table I). This is due to a lower plasma density in the conventional RIE system. Furthermore, for the conventional RIE system, the etch selectivity between the SWNT film and the resist mask has decreased to 1:2.8, 1:3.8, and 1:11 for S1813, LOR3B, and PMMA, respectively. This is due to a reduction in the physical etching component using the conventional RIE system. These results demonstrate that the use of an ICP etcher provides significant advantages, such as faster etch rates and better selectivity, over conventional parallel plate plasma systems in order to be able to pattern submicron features in nanotube films.

Using the initial etch recipe parameters, we have fabricated standard four point probe structures with the three types of resist processes in order to characterize the effect of the process chemistry on the resistivity of the patterned nanotube films. Figure 5 shows an optical microscope image of a typical four point probe structure we have fabricated with 50 μm width and 750 μm length. For some of the four point probe devices, such as the one shown in Fig. 5, chromium/palladium metal contacts were patterned on the nanotube film pads by photolithography (using the S1813 only resist process), e-beam evaporation, and subsequent lift-off. Identical resistivity values were measured regardless of whether the electrical probes were placed on the Cr/Pd metal contacts or directly on the nanotube film pads since the effects of contact resistance are eliminated in a four point probe measurement. The resistivity values reported in this article were obtained from four point probe structures which did not have the metal contacts. This enables us to compare the effects of the three types of resist processes on the resistivity of nanotube films directly, without introducing any contamination due to the second lithography process used for metal contact patterning.

We have recently observed that the resistivity of patterned nanotube films increase significantly compared to bulk films as their width and thickness shrink, particularly for devices having submicron dimensions. Therefore, for this experiment, we designed structures with large length (L=500 and 1000 μm), large width (W=10, 20, 30, 50, and 100 μm), and large thickness (t=75 nm) to avoid geometrical effects on the resistivity of patterned nanotube films. In other words,
the resistivity values measured using these large structures are the minimum resistivity values representative of those of bulk SWNT films.27

After fabrication, the devices were measured at room temperature and in ambient atmosphere. Current was applied between the outer pads (labeled 1 and 2 in Fig. 5), and the voltage drop between the two inner pads (labeled 3 and 4 in Fig. 5) was measured. The resistivity \( \rho \) of each structure was obtained from the usual formula,

\[
R = \rho (L/Wt),
\]

where \( R \) is the resistance equal to the slope of the measured linear \( I-V \) curves. The resistivity values obtained for the ten devices of different \( L \) and \( W \) dimensions fabricated by the same resist process were found to agree within 5\%. Table II lists the average resistivity values measured using four point probe structures patterned by the three different resist processes, showing that the LOR3B/S1813 dual layer resist process results in the highest resistivity, followed by the PMMA/S1813 process, with the S1813 only process giving the lowest resistivity. Furthermore, for all three resist processes, the resistivity values are higher than those measured for the as-prepared nanotube film, which is about \( 1.5 \times 10^{-4} \) \( \Omega \) cm.32 It is known that nitric acid, which is used for purifying SWNTs, dope them and hence decreases the film resistivity during nanotube film preparation.5 The increase in resistivity after lithography is likely due to the partial dedoping of the acid purified nanotubes during the processing steps associated with the patterning. The results in Table II suggest that the level of dedoping depends on the resist chemistry, yielding different resistivity values for different resist processes.

The fabricated four point probe structures were also used to investigate the effect of ICP reactive ion etching on the resistivity of partially etched nanotube films. Nanotube film devices with an initial thickness of 100 nm were etched by the initial recipe for 10 s (so that the film becomes about \( \sim 24 \) nm thinner), and the resistivity of the film before and after etching was compared. Regardless of the size and location of the devices, their resistivity increased between two to three orders of magnitude, showing that the remaining film has been significantly damaged by the \( O_2 \) plasma during etching. Since the nanotube film is porous, reactant species can penetrate into the film and can damage the nanotubes deeper in the film. Nanotubes that have been damaged can no longer contribute to electrical conduction, and therefore the film resistivity increases significantly.

<table>
<thead>
<tr>
<th>Resist process</th>
<th>S1813 only</th>
<th>PMMA/S1813 dual layer</th>
<th>LOR3B/S1813 dual layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistivity ( (10^{4} ) ( \Omega ) cm)</td>
<td>5.2</td>
<td>6.3</td>
<td>6.9</td>
</tr>
</tbody>
</table>

IV. CONCLUSIONS

In conclusion, we have demonstrated the ability to efficiently pattern SWNT films with good selectivity and directionality down to submicron lateral dimensions by photolithography or e-beam lithography and \( O_2 \) plasma etching using an ICP-RIE system. We systematically studied the effect of ICP-RIE etch parameters on the nanotube film etch rate and etch selectivity. Decreasing the substrate power from 100 to 15 W decreased the nanotube film and resist etch rates by about a factor of 10. Decreasing the chamber pressure from 45 to 10 mTorr increased the nanotube film and resist etch rates by a factor between 1.7 and 3.5. It also increased the etch selectivity between the nanotube film and the resist masks. On the other hand, increasing the chamber pressure from 45 to 100 mTorr did not change the etch rates of the nanotube film and resists significantly. Similarly, increasing the helium flow rate (which actively cools the substrate) from 10 to 40 sccm did not produce a significant change on the etch rates of the SWNT film and the three resists. Furthermore, the SWNT film etch rate was found to be independent of the linewidth etched for linewidths ranging from 50 \( \mu \)m down to 100 nm.

By fabricating standard four point probe structures using the patterning capability developed, we have also shown that different resist processes result in different SWNT film resistivity values due to partial dedoping of the acid purified nanotubes during lithography. In addition, the resistivity of nanotube films increased between two to three orders of magnitude after partial etching by the \( O_2 \) plasma, indicating that the remaining film is significantly damaged during the etch.

In addition, by comparing the etch rates of the SWNT film and the three resists in an ICP-RIE system to those in a conventional parallel plate RIE system, we have demonstrated that using an ICP-RIE system provides significant advantages, such as faster etch rates and better etch selectivity, over conventional parallel plate RIE plasma systems, making it possible to pattern lateral features as small as 100 nm in nanotube films. Furthermore, we have shown that a wide range of nanotube film etch rates can be obtained using an ICP-RIE system by changing the substrate bias power and chamber pressure.

In short, the nanolithographic patterning capability developed and systematically investigated in this work could open up significant opportunities for fabricating and integrating single-walled nanotube films into a wide range of electronic and optoelectronic devices.

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