Video Article

Precision Milling of Carbon Nanotube Forests Using Low Pressure Scanning Electron Microscopy

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Abstract

A nanoscale fabrication technique appropriate for milling carbon nanotube (CNT) forests is described. The technique utilizes an environmental scanning electron microscope (ESEM) operating with a low pressure water vapor ambient. In this technique, a portion of the electron beam interacts with the water vapor in the vicinity of the CNT sample, dissociating the water molecules into hydroxyl radicals and other species by radiolysis. The remainder of the electron beam interacts with the CNT forest sample, making it susceptible to oxidation from the chemical products of radiolysis. This technique may be used to trim a selected region of an individual CNT, or it may be used to remove hundreds of cubic microns of material by adjusting ESEM parameters. The machining resolution is similar to the imaging resolution of the ESEM itself. The technique produces only small quantities of carbon residue along the boundaries of the cutting zone, with minimal effect on the native structural morphology of the CNT forest.

Video Link

The video component of this article can be found at <https://www.jove.com/video/55149/>

Introduction

Carbon nanotubes (CNTs) and graphene are carbon-based nanomaterials that have attracted significant attention because of their superior strength, durability, thermal, and electrical properties. Precision machining of carbon nanomaterials has become an emerging topic of research and offers the potential to engineer and manipulate these materials towards a variety of engineering applications. Machining CNTs and graphene requires nanoscale spatial precision to first locate a nanoscale area of interest and then to selectively remove only the material within the area of interest. As an example, consider the machining of vertically oriented CNT forests (also known as CNT arrays). The cross section of CNT forests may be precisely defined by lithographic patterning of catalyst films. The top surface of the vertically oriented forests, however, are frequently poorly ordered with non-uniform height. For surface-sensitive applications such as thermal interface materials, the irregular surface may impede optimal surface contact and reduce device performance. Precision trimming of the irregular surface to create a uniform flat surface could potentially offer better, more repeatable performance by maximizing the available contact area.

Precision machining techniques for nanomaterials frequently do not resemble conventional macroscale mechanical machining technologies such as drilling, milling, and polishing by means of hardened tooling. To date, techniques using energetic beams have been most successful at site-selective milling of carbon nanomaterials. These techniques include laser, electron beam, and focused ion beam (FIB) irradiation. Of these, laser machining techniques provide the most rapid material removal rate^{1,2}; however, the spot size of laser systems is on the order of many microns and is too large to isolate nanometer-scale entities such as a single carbon nanotube segment within a densely populated forest. By contrast, electron and ion beam systems produce a beam that may be focused to a spot that is several nanometers or less in diameter.

FIB systems are specifically designed for nanoscale milling and deposition of materials. These systems utilize an energetic beam of gaseous metal ions (typically gallium) to sputter material from a selected area. FIB milling of CNTs is achievable, but often with unintended byproducts including gallium and carbon redeposition in surrounding regions of the forest^{3,4}. When the technique is used for CNT forests, the redeposited material masks and/or alters the morphology of selected milling region, altering the native appearance and behavior of the CNT forest. The gallium may also implant within the CNT, providing electronic doping. Such consequences often make FIB-based milling prohibitive for CNT forests.

Transmission electron microscopes (TEMs) utilize a finely focused beam of electrons to probe the internal structure of materials. Acceleration voltages for TEM operation typically range from 80-300 kV. Because the knock-on energy of CNTs is 86.4 keV⁵, the electron energy produced by TEM is sufficient to directly remove atoms from the CNT lattice and induce highly localized milling. The technique mills CNTs with potentially sub-nanometer precision^{5,6,7}; however, the process is very slow – often requiring minutes to mill a single CNT. Importantly, TEM-based milling approaches require CNTs to first be removed from a growth substrate and dispersed onto a TEM grid for processing. As a result, TEM-based methods are generally not compatible with CNT forest milling in which the CNTs must remain on a rigid substrate.

Milling of CNT forests by scanning electron microscopes (SEMs) has also received attention. In contrast to TEM-based techniques, SEM instruments are typically unable to accelerate electrons with sufficient energy to impart the knock-on energy required to directly remove carbon atoms. Rather, SEM-based techniques utilize an electron beam in the presence of a low-pressure gaseous oxidizer. The electron beam selectively damages the CNT lattice and may dissociate the gaseous ambient into more reactive species such as H_2O_2 and the hydroxyl radical. Water vapor and oxygen are the most commonly reported gases to achieve selective area etching. Because the SEM-based techniques rely on a multiple-step chemical process, numerous processing variables may influence the milling rate and precision of the process. It has been previously observed that increasing acceleration voltage and beam current directly increase the milling rate because of an increased energy flux, as expected¹¹. The effect of chamber pressure is less obvious. A pressure that is too low suffers from a deficiency of the oxidizing agent, decreasing the milling rate. Further, an over-abundance of gaseous species scatters the electron beam and decreases the electron flux in the milling region, also decreasing the material removal rate.

To estimate the carbon removal rate, an approach similar to that used by Lassiter and Rack¹² was employed, whereby electrons interact with precursor molecules near the surface to generate reactive species that etch the substrate surface. From this model, the etch rate is estimated as

$$\text{Etch Rate} \approx \frac{N_A}{Z} x A_\sigma \Gamma_e$$

where N_A is the surface concentration of the etchant species, Z is the surface concentration of available reaction sites, x is a stoichiometry factor relating the volatile etching products generated relative to the reactants, A_σ represents the probability of generating the desired etching species from an electron-water vapor collision, and Γ_e is the electron flux at the surface. The factors of x and A_σ are assumed to be unity, while Z is assumed to be nearly constant and significantly larger than N_A . Further details may be found in our previous work.¹¹

In this article, a procedure is explored that uses low-pressure water vapor within a SEM to mill regions ranging from individual CNTs to large volume (tens of cubic micrometers) material removal. Here we demonstrate the technique used to mill CNT forests using an ESEM by the use of reduced area rectangles, horizontal line scans, and software-controlled rastering of the electron beam. Additional software and hardware are required for pattern generation, as outlined in the Materials List. Emphasis is placed on removing relatively large (100's of cubic microns) material volume from a CNT forest, so the following processing conditions are relatively aggressive.

When handling the sample and the sample stub, it is important to wear disposable nitrile gloves. This will prevent oils from being transferred to the stub or sample and consequently deteriorating the effectiveness of the pumps.

Protocol

1. Preparation of CNT Forest Sample for Milling

1. CNT Synthesis

1. Deposit 10 nm of aluminum oxide (alumina) on a thermally oxidized silicon wafer using atomic layer deposition¹³ or other physical vapor deposition methods.
2. Deposit 1 nm of iron on the alumina support layer by sputtering¹⁴ or other physical vapor deposition method.
3. Synthesize CNTs using an established process, such as thermal chemical vapor deposition¹⁵.
 1. Heat a 20 mm diameter tube furnace to 750 °C in 400 standard cubic centimeters (sccm) of flowing helium and 100 sccm hydrogen. Introduce 100 sccm ethylene as a hydrocarbon feedstock gas for a growth rate of approximately 50 $\mu\text{m}/\text{min}$.

2. SEM Preparation

1. Apply carbon tape to a standard 1/2" diameter SEM stub. If tilting the stage is required, overlap the region of the CNT forest sample to be milled over the edge of the stub. If software-controlled electron beam rastering will be used in the milling procedure, secure the CNT sample to an electron beam lithography mount in a similar manner.
2. If milling the CNT cross section, secure the stub to a 45° stub holder with a set screw.
3. Vent the ESEM by selecting the "Vent" icon from the ESEM control software.
4. Open the ESEM stage door, and secure the stub to the SEM stage with a set screw.
5. Close the SEM chamber and select "High Vacuum" in the ESEM control software.
6. While the ESEM chamber is pumping, select the electron beam parameters of 5 kV and spot size of 3.0 using the Beam Control tab within the control software.
7. Select the secondary electron detector by selecting Detectors | ETD(SE) in the ESEM control software.
8. Select the "Beam On" icon in the control software. The beam can be activated only once the chamber vacuum is less than 10^{-4} Torr. Use manual SEM focus control knobs to focus the sample.
9. Tilt the sample to 45° using manual tilt stage control knob or by inputting 45° in the "Tilt" field in the "Coordinates" tab of the ESEM software. Focus on the highest sample. Link the focal distance to the working distance by selecting Stage | Link Z to FWD in the ESEM software menu. Input 7 mm into the "Z" field in the "Coordinates" tab within the control software.
10. Adjust focus, stigmatism, brightness and contrast using the manual control knobs to resolve a well-focused image.

3. Beam Adjustment in High Vacuum Mode

1. Locate a region for milling using navigation controls. Double click within the SEM image view or by manually turning the x and y control knobs of the SEM stage control to navigate.
2. Navigate to an adjacent location approximately 100 μm away from the milling region.
3. Consult **Figure 1** to estimate the material removal rate of the CNT forest as a function of pressure, acceleration voltage, dwell time per pixel, and beam current.

4. Adjust the acceleration voltage to 30 kV and spot size to 5.0 using the ESEM control software. Adjust image focus, brightness, and contrast using the ESEM control knobs. For nanometer-scale milling of individual or few CNTs, select 5 kV and spot size of 3.0.
 5. Select a 1 mm aperture by manual aperture adjustment. Adjust focus, stigmation, brightness, and contrast to obtain a well-resolved image, as previously detailed.
 6. Decrease magnification to <1,000X.
- 4. SEM Setup in Low Pressure Water Vapor**
1. Select a pressure of 11 Pa in the control software dropdown box.
 2. Select "Low Pressure" mode in the "Vacuum" settings in the ESEM software to introduce water vapor.
 3. Select "Beam On" in the control software upon pressure stabilization. Select a dwell time of <10 μ s and a resolution of 1,024 x 884 in the drop-down boxes of the control software.
 4. Adjust the image brightness, contrast, focus, and stigmation as previously detailed.
 5. Navigate to the desired milling region. Rotate the image orientation by selecting Scan | Scan Rotation in the control software, if required. Select a suitable rotation angle that aligns with the native vertical and horizontal scan orientation of the SEM.
 6. For milling feature sizes on the order of 1 μ m, select a magnification of 40,000X. Select a magnification of 20,000X to mill features with dimensions up to 5 μ m.
 7. Pause the electron beam by selecting the ' ' icon . An image of the CNT forest will be displayed and may be used for selecting reduced area milling regions while the beam is paused.

2. CNT Forest Milling

- 1. Instructions for CNT forest milling using a rectangular selected area**
 1. Choose the 'Reduced Area' tool in the control software, or select Scan-Reduced Area in the software menu. Extend a reduced area rectangle over the area to be milled.
 2. Adjust the image resolution to 2,048 x 1,768. Increase the dwell time to 2 ms. If 2 ms is not available, navigate to the Scan | Preferences and select the "Scanning" tab. Select an existing scan time and type "2.0 ms" into the "Dwell Time" field. Click "OK" to close the menu.
 3. Select the ' ' icon in the control software to activate the electron beam.
 4. Select the ' ' icon so that the beam rasters over the selected area one time. Select the icon immediately after step 2.1.3. The scan duration depends on the size of the selected area, resolution and dwell time and may be approximated by multiplying the number of pixels within the scan area and the dwell time per pixel.
 5. Decrease magnification to <1,000X once the beam has completed rastering the selected area. Revert to the parameters used in step 1.3, including High Vacuum. Select "Beam On" to engage the beam.
- 2. Instructions for CNT forest milling along a horizontal line**
 1. Select the line scan feature by navigating to Scan | Line in the control software. The line width is determined by the size of the electron beam itself. Adjust the image resolution to 2,048 x 1,768 from the control software dropdown box. Increase the dwell time to 2 ms as detailed in step 2.1.2.
 2. Using the still image acquired before pausing the electron beam, place the line over the area to be milled.
 3. Select the videoscope icon or navigate to the Scan menu and select "Videoscope." Using the videoscope tool provides feedback relative to when a line scan has fully completed.
 4. Select the ' ' icon to scan electron beam across the width of the line.
 5. Select the ' ' icon to blank the electron beam.
- 3. Instructions for CNT Forest milling using software-controlled electron beam rastering**
 - 1. Pattern Generation**
 1. Design a milling pattern of interest using a CAD software package such as AutoCAD.
 2. Using "Nanometer Pattern Generation System" (NPGS) software, import the CAD pattern file.
 3. Convert the shapes to solid features by selected "Filled Polygons" in the NPGS software.
 4. Save the drawing as a '.dc2' file in a designated project folder of NPGS.
 5. Using NPGS, navigate to the project folder containing the ".dc2" file. Right select the ".dc2" file and select "Run File Editor" to convert the drawing to NPGS code. Typical parameters used to pattern CNT forests at given conditions are as listed below:
Center-to-center distance = 5 nm
Line Spacing = 5 nm
Magnification = 10,000X
Desired Beam Current = 26
Line Dose = 100 nC/cm
 - 2. Electron Beam Milling using NPGS Lithography Software**
 3. Select the "NPGS Mode" in NPGS software button to give control of the SEM to NPGS.
 4. Highlight the pattern file and select "Process Run File" in NPGS to initiate milling.
 5. Select "SEM Mode" in the NPGS software when patterning is finished. Select "High Vacuum" in the ESEM control software.
 6. Select "Beam On" to inspect the milled region. Use conditions detailed in step 1.3.

3. Sample Removal

1. Vent the chamber by selecting "Vent" in the ESEM control software.

2. Open the ESEM door. Remove the stub by loosening the set screw.
3. Close the chamber door. Select "High Vacuum" in the control software.

Representative Results

The ESEM technique was used to mill a CNT forest synthesized using thermal CVD^{15,16}. Selected area removal of a few CNTs from within a forest is shown in **Figure 2**¹¹. For this demonstration, parameters include 5 kV, spot size of 3, 11 Pa, 170,000X magnification, 2 ms dwell time, and an aperture of 30 μm .

To demonstrate a larger-scale area removal, the top surface of a CNT forest micropillar was selected for milling. SEM conditions are selected for rapid, large-area CNT forest removal. Namely, these conditions include a magnification of 20,000X, a pressure of 11 Pa, acceleration voltage of 30 kV, spot size of 5, dwell time of 2 ms, and a 1 mm aperture setting. A reduced area box is chosen such that the irregular top surface to be removed is enclosed within the selected area. SEM micrographs of the CNT forest pillar are shown in **Figure 3** before and after the selective area milling process. The red line in the figure represents the lower bound of the reduced area box used for milling.

Non-rectangular geometries achieved using software-controlled electron beam rastering and a relatively short 20 μm tall CNT forest. As shown in **Figure 4**, a 15 μm diameter circle was machined into a CNT forest. For this demonstration, the CNT forest was milled parallel to the CNT growth direction (normal to the substrate). Milling parameters used for this demonstration include a magnification of 10,000X, a pressure of 11 Pa, acceleration voltage of 30 kV, spot size of 5, dwell time of 2 ms, and 1 mm aperture setting. **Figure 4** shows that the process milled the CNTs fully to the underlying silicon substrate.

Figure 1: Material Removal Rate Variation. Material removal rate (MRR) variation. SEM micrographs demonstrate the MRR in the transverse direction (a) by varying operating pressure from 133, 66, 33, 66, and 11 Pa (top to bottom) and (b) in the axial cutting direction by varying dwell time from 3, 2, 1, and 0.5 ms/pixel (left to right). The MRR is plotted as a function of incremental changes in pressure, acceleration voltage, beam current, and dwell time in the (c) transverse and (d) axial cutting directions. The MRR as a function of electron dose varies nearly linearly in both the (e) transverse and (f) axial milling orientation. This figure is reproduced with permission from reference¹¹. [Please click here to view a larger version of this figure.](#)

Figure 2: Milling of Individual CNTs. SEM micrograph showing individual CNTs from within a forest selected for local milling (a) before and (b) after milling. This figure is reproduced with permission from reference¹¹. [Please click here to view a larger version of this figure.](#)

Figure 3: Milling of a CNT Forest. A 10 μm wide CNT forest pillar (a) before and (b) after selective area milling using ESEM-based milling. Milling conditions include magnification of 20,000X, a pressure of 11 Pa, acceleration voltage of 30 kV, spot size of 5, dwell time of 2 ms per pixel, and 30 μm aperture. The red line in the figure represents the lower bound of the selective area rectangle used in the milling process. [Please click here to view a larger version of this figure.](#)

Figure 4: Patterned Milling of CNT Forest. Software-controlled electron beam rastering is used to define and mill a 15 μm diameter circle in a CNT forest. In this setup, the milling direction was parallel to the CNT growth direction from the top surface to the underlying substrate. [Please click here to view a larger version of this figure.](#)

Figure 5: Deposited Carbon after Milling. SEM micrographs showing the surface finish of ESEM milled CNT forests. (a) The top surface of a CNT forest shows the surface variation between the milled and as-synthesized regions. (b) Higher magnifications reveal that some amorphous carbon deposits are left behind during the cutting process. [Please click here to view a larger version of this figure.](#)

Discussion

The protocol details best practices for milling relatively large (micron-scale) features in CNT forests. In general, the material removal rate may be reduced by reducing the acceleration voltage, spot size, and aperture diameter. To trim a specific CNT within a forest, recommended conditions include 5 kV, a spot size of 3, and an aperture that is 50 μm or less in diameter. Note that the milling technique using reduced area rectangles is detailed such that the electron beam rasters the enclosed region only one time. The reduced area may be scanned multiple times if additional cutting depth is desired; however, we illustrate a single scan for simplicity. We note that the extended electron beam dwell time, high current, and high acceleration voltage represent conditions that are frequently avoided for imaging of carbon-based materials; however, these aggressive parameters in a low-pressure water vapor ambient are critical for achieving large-scale milling. Further, we note that similar imaging conditions in the absence of low-pressure water vapor results in little CNT damage.

The ESEM-based milling method described in this work is a minimally disruptive machining method that preserves neighboring CNT forest structural morphology. The technique is amenable to removing nanoscale features such as segments of individual CNTs and also to removing regions spanning many microns. We demonstrate the technique using reduced area rectangles, lines, and arbitrary patterns using software-controlled electron beam rastering. While the technique is relatively clean compared to FIB-based milling, small amounts of carbon residue exists on milled surfaces. Current research is addressing avenues for reducing this residue. Additionally, the material removal rates shown in **Figure 1** were obtained for a CNT forest with CNTs featuring an average outer and inner diameter of 10 and 7 nm, respectively. Material removal rates are expected to be a function of CNT density, CNT diameter, and CNT alignment. **Figure 1** should be consulted as a guide, recognizing that the stated material removal rate is specific to this CNT forest morphology. While qualitative trends represented in the figure are expected to hold for all CNT forests, some experimentation may be required to find the optimal parameters for a different material system.

While the ESEM machining methodology is demonstrated using CNT forests, it is equally applicable for graphene and other carbon-based materials. The technique does not require delamination of the CNT forest for processing and does not introduce external heavy elements that may significantly alter the surrounding CNT forest morphology. The procedure may be used for inspecting CNT forest internal morphology, and perhaps for producing 3-D lattices structures for microscale prototyping which may be functionally coated (with alumina for enhanced stiffness^{17,18}, for example).

The technique is currently being utilized to examine the internal structural morphology of CNT forests. Because structural morphology is intimately linked with functional properties^{16,19,20,21,22}, characterization of CNT forest morphology in three dimensional space may provide additional insights into the governing structure-property relationships. With the ability to precisely mill into a forest and observe the internal nanotube interactions, CNT forest synthesis modeling and analytical models can be tuned and validated.

The emphasis of the ESEM milling technique to date has been directed towards rapid material removal with less focus on optimizing conditions for reducing the residual carbon residue. A future direction is to explore the mechanism of amorphous carbon deposition in the immediate vicinity of the cut surfaces when large volumes of materials are removed, as shown in **Figure 5**. With a broad parameter space available for exploration, including environmental gas composition, vapor pressure, acceleration voltage, probe current, and electron beam rastering conditions, enhanced surface cleanliness may be achieved.

Disclosures

The authors declare that they have no competing financial interests.

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