

Engineering Multiwalled Carbon Nanotubes Inside a Transmission Electron Microscope Using Nanorobotic Manipulation

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Abstract—This paper provides a review of recent experimental techniques developed for shell engineering individual multiwalled carbon nanotubes (MWNTs). Basic processes for the nanorobotic manipulation of MWNTs inside a transmission electron microscope are investigated. MWNTs, bamboo-structured carbon nanotubes (CNTs), Cu-filled CNTs, and CNTs with quantum dots attached are used as test structures for manipulation. Picking is realized using van der Waals forces, “sticky” probes, electron-beam-induced deposition, and electric breakdown. Cap opening and shell shortening are presented using field emission current. Controlled peeling and thinning of the shells of MWNTs are achieved by electric breakdown, and changes in MWNT structures are correlated with electrical measurements. These processes are fundamental for the characterization of nanoscale materials, the structuring of nanosized building blocks, and the prototyping of nanoelectromechanical systems.

Index Terms—Carbon nanotubes (CNTs), nanorobotic manipulation, shell engineering, transmission electron microscope (TEM).

I. INTRODUCTION

IN RECENT years, nanorobotic manipulation in SEMs [1]–[3] has been demonstrated as a powerful enabling technique for fabricating nanoelectromechanical systems (NEMS) [4] from nanomaterials and structures. Systems and processes have been developed for manipulating, structuring, characterizing, and assembling as-grown nanomaterials and as-fabricated nanostructures [3], [5]. With relatively high resolution and large-specimen chambers, an SEM can provide nanorobotic manipulators with an excellent environment for real-time observation and complex manipulation. However, the best resolution of a high-grade commercially available SEM is typically around 1–2 nm in an ideal environment. The nominal achievable resolution

in a real SEM environment, however, is usually only several nanometers because of base vibration and other types of noise. Though this is not satisfactory for high-resolution investigations, transmission electron microscopes (TEMs) typically have subnanometer to atomic resolution. Nanomanipulation can also benefit from the nature of electron transmission and real-time imaging of a TEM. However, the narrow vacuum chamber and the thin specimen holder present a challenge for developing nanomanipulators that operate inside TEMs. This has greatly limited the use of nanorobotic manipulation in TEMs as compared to SEMs or scanning probe microscopes (SPMs).

Previous investigation on nanomanipulation in a TEM has adopted stack or tube piezoactuators built in a TEM specimen holder for positioning a probe against a specimen [6], [7]. Some of these holders have coarse motion being realized either with a stepping motor or by manual mechanical adjustment via a feedthrough. Without a motion amplifier, a stack PZT or piezotube actuator can typically output motion in the range of several micrometers to several tens of micrometers, which makes the manipulation difficult because the operator has to put the samples into the micrometer-scale working space of the manipulator manually, though for 1 or 2 DOF, coarse motion can be helpful. Hence, early successful experiments have mostly been based on specially prepared specimens such as preconnected samples or arrays in order to simplify the alignment of the probe and sample or to have them initially aligned. To solve the manipulation problem, a hybrid approach [8] has been investigated by fine-positioning samples on a TEM manipulator inside an SEM with an SEM manipulator. For this purpose, a passive TEM sample holder has been introduced, which can be deformed plastically so that the final sample preparation can be done in an SEM before inserting the TEM holder back into a TEM for the actual fine manipulation. Although this is an effective method, the *ex situ* nature of this approach necessitates the moving of the holder between SEM and TEM, thus significantly decreasing the success rate. Recent improvements of actuators create more promising strategies. An example is a scanning tunneling microscope (STM) placed on a TEM holder (such as the commercially available ST1000 STM–TEM holder from Nanofactory). By adopting an ST1000, we are able to systematically investigate the basic manipulation techniques for handling individual carbon nanotubes (CNTs) in a TEM. Tools and basic processes for *in situ* manipulation, structuring, characterization, and assembly of individual nanotubes have been created for prototyping nanotube-based NEMS.

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Controllable exposure of the shells of multiwalled CNTs (MWNTs) [9]–[15] has evoked interest because of the possibility of their application in NEMS such as ultralow-friction bearings [14], [16], gigahertz nanooscillators [17], [18], and nanometer-scale actuators [19]. *In situ* manipulation of the nanotube core allows controlled reversible telescoping motion and, furthermore, the associated forces to be quantified [1]. The steady-state resistance to interlayer sliding motion has been measured to be 0.08–0.3 MPa [20]. Robust ultralow-friction linear nanobearings and rotary microactuators have been demonstrated on the basis of interlayer rotation of an MWNT [21]–[23]. Open-ended MWNTs have been created by removing the commonly capped ends with acid etching [24], saturated current [25], electric pulse [9], or mechanical strain [11], thus providing access to inner-core nanotube cylinders. Acid etching is effective for opening nanotube caps but does not expose inner layers in a controlled way, whereas electric pulse and mechanical strain are convenient *in situ* processes for atomic level imaging and property characterization in a TEM, electric breakdown with saturated current is potentially a large-scale manufacturing method.

In this paper, the system setup is introduced in Section II. Basic processes for manipulation are then presented in Section III. Newly developed picking up processes using “sticky” probes and electric breakdown are presented and applied to various CNT-based structures including bamboo-structured CNTs, CNTs with quantum dots (QDs) attached, and Cu-filled CNTs besides as-synthesized CNTs. Current-driven shell engineering of MWNTs is shown in Section IV. Breakdown of individual nanotube shells is extended for the peeling of the innermost layers and the shrinking of a single layer.

II. SYSTEM SETUP

The ST1000 STM–TEM holder (Nanofactory) has an STM unit built in a TEM sample holder [Fig. 1(a)]. The specimen area of the sample holder is shown in Fig. 1(b). The sample is conductively glued to a piece of gold wire (0.35 mm diameter) in a specimen holder, while the gold wire (0.25 mm diameter) STM tip is mounted in a tip holder hemisphere with six elastic legs connected to a piezoscanner via a sapphire ball, which allows both millimeter-scale coarse sliding and subnanometer fine movement in all three dimensions [X -, Y -, and Z -directions, as shown in Fig. 1(a)]. In the coarse-motion mode, the tip holder is actuated in a stick-slip inertial sliding way, whereas in the fine-motion mode, the tip holder is positioned by the solid deformation of the piezoactuators from the same manipulator. The superposition of the coarse and fine motions in the same mechanism makes the manipulator compact enough to be contained in a TEM holder. To get subnanometer resolution for both manipulation and *in situ* observation, the holder is mounted inside a Phillips CM30 TEM equipped with a goniometer [Fig. 1(c)].

Besides the standard 0.25-mm-etched golden wire probes, we have developed a set of special tools for different purposes. Tungsten probes [Fig. 2(a) and (b)] are prepared by attaching the 10 μm tip of a commercially available tungsten probe (Pico-probe, T-4-10-1 mm, tip radius < 100 nm) onto a golden wire with silver cement. These probes have a higher hardness and are more suitable for manipulation than the golden wires designed

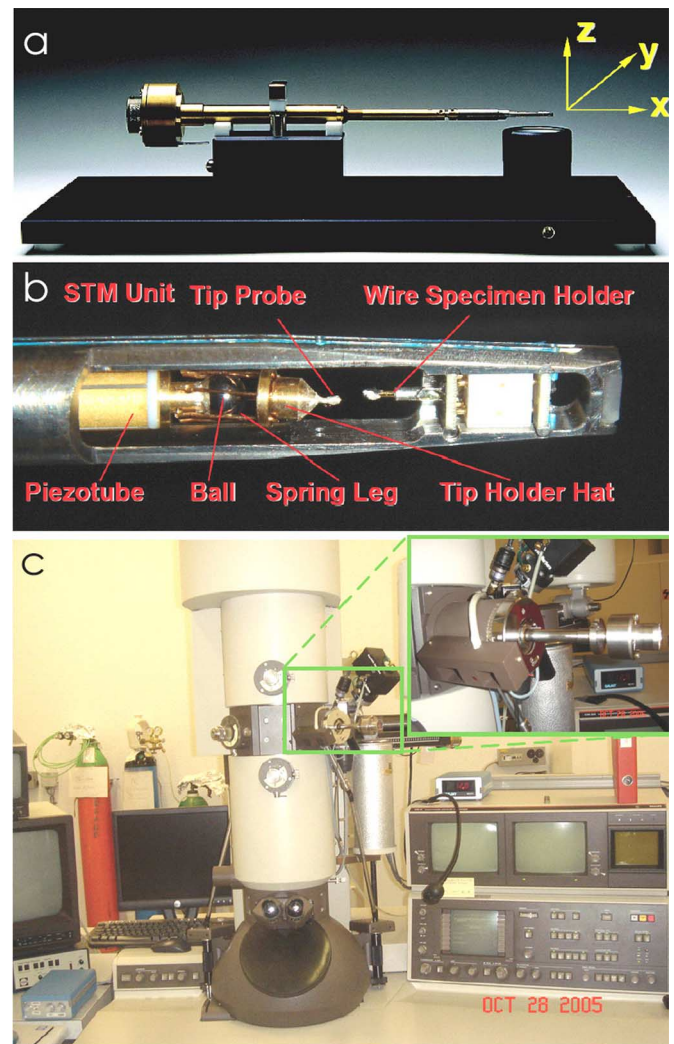


Fig. 1. Nanorobotic manipulation system in a TEM. (a) ST1000 STM–TEM holder (Nanofactory). (b) STM unit. (c) Installation of the STM–TEM holder in a Philips CM30 TEM.

for STM imaging. Dipping these probes into a double-sided silver conductive tape (Ted Pella, Inc.) for SEM, “sticky” probes are prepared [Fig. 2(c) and (d)], which are suitable for picking up relatively large objects. Hooks [Fig. 2(e) and (f)] are formed by controlled “tip-crash” of the thin tungsten probes onto a substrate. Atomic force microscopy (AFM) cantilevers [Fig. 2(g) and (h)] are prepared for force measurement. These are commercially available AFM cantilevers that have been modified by cleaving the rear part from the chip and attaching it onto a 0.35-mm Au wire with silver paint, which is, in turn, installed onto the wire specimen holder. These special tools greatly extend the applicable range of the STM–TEM holder. They can also be shared with different manipulators in an SEM and a TEM, making it possible to investigate the same sample in different environments. In this paper, we show the application of the tungsten probes and the “sticky” probes.

III. BASIC PROCESSES

Basic processes have been developed for manipulating and structuring individual CNTs. MWNTs synthesized from arc

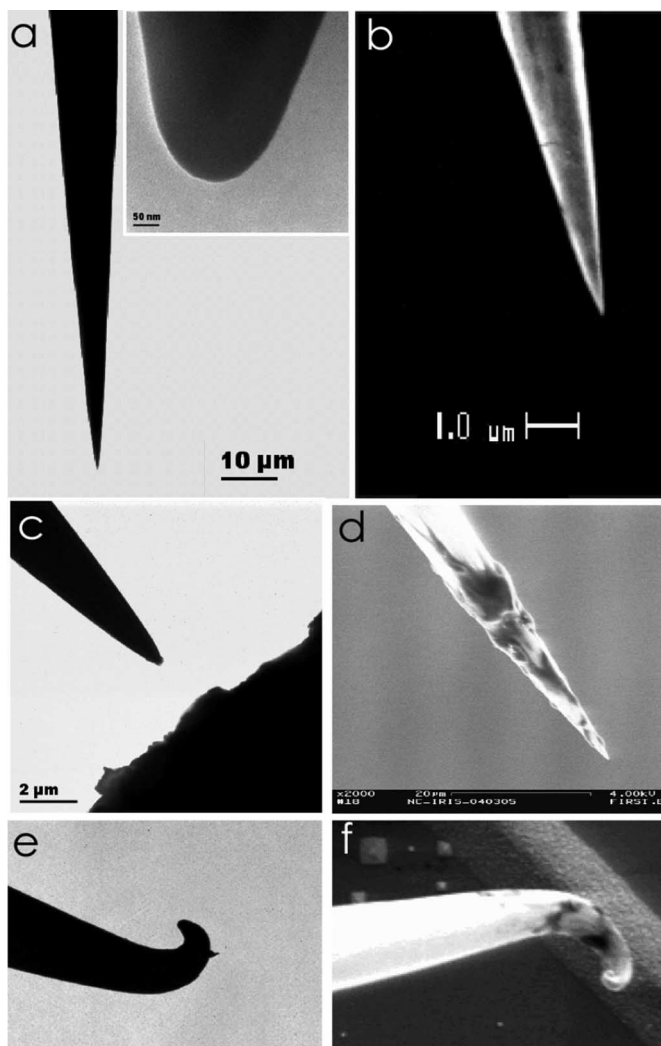


Fig. 2. Special tools. (a) and (b) TEM and SEM images of tungsten probes [inset of (a) shows the tip]. (c) and (d) TEM and SEM images of “sticky” tungsten probes. (e) and (f) TEM and SEM images of nanohooks. (g) and (h) TEM and SEM images of AFM cantilevers.

discharge and CVD, and Cu-filled MWNTs from thermal CVD [26] are used to show the universality of the processes.

A. Picking Up Specimens

Picking up specimens is the most important and fundamental process for 3-D manipulation and assembly, and in most cases, the most difficult process also. The essential procedures include: 1) alignment of a tool, typically a probe, with an object and 2) exertion of a force between the probe and the object larger than that between the object and a substrate supporting it. In an SEM, dielectrophoresis, van der Waals forces, and electron-beam-induced deposition (EBID) have been shown to be effective [3].

In a TEM, different challenges exist. Because of the nature of the transmission imaging, it is more difficult to know the relative position between the probe and the sample along the electron beam direction. When a dense, and as a result, nontransparent probe is adopted, the situation is even worse. A TEM is

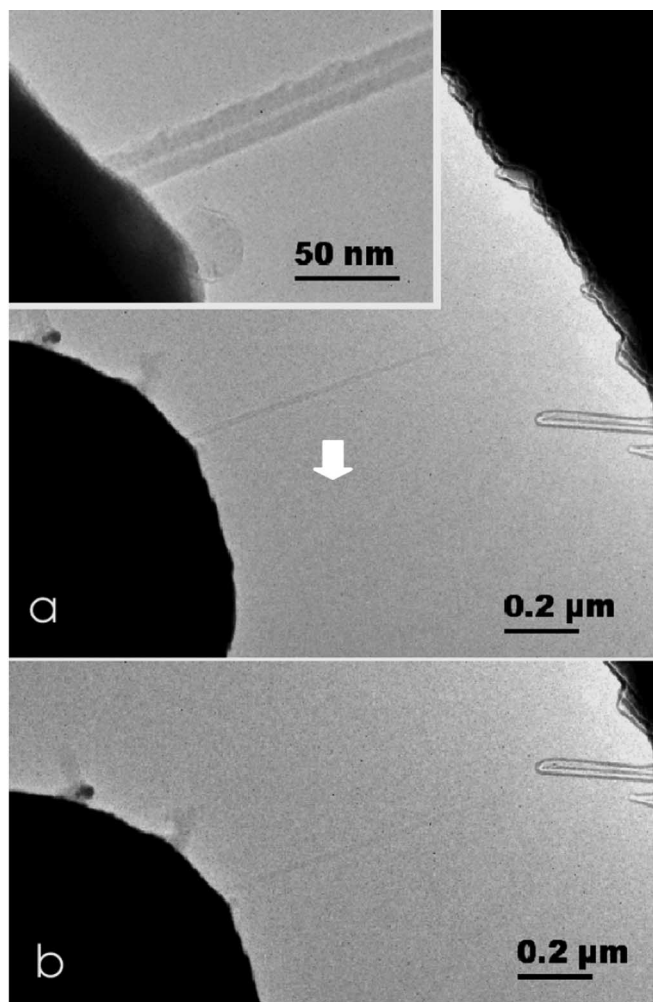


Fig. 3. Picking up a nanotube using van der Waals force and moving it downward from the position shown in (a) to (b). Inset shows the contact part with higher magnification. The contact point is hidden by the probe.

generally equipped with apparatus to protect the samples from contamination for typical contaminants such as pump oil. This makes EBID based on such contaminants [27] difficult to be performed. For tackling these new challenges, we have used “wobble” focusing to position the probe relatively far from the focusing plane of the sample. We then approach the sample with the probe from the same side until a contact is established. The probe is biased with a low voltage (typically 100 mV) so that the contact can be detected from the current readout of an ammeter. Another strategy is based on the instant deformation of a nanotube at the moment of a contact.

The fixation of a nanotube onto a probe can be realized with van der Waals forces, EBID or sticky materials. Because of the decreased contamination, surfaces of probes and nanotubes stay generally clean for a longer time, which enhances the van der Waals interaction between them. Fig. 3 shows an MWNT being picked up with a probe (a) after fixation over EBID (inset) and then being moved to a different position (b).

EBID can be used to fix a nanotube onto a probe, but deposition takes much longer in a TEM than in an SEM. Fig. 4(a)

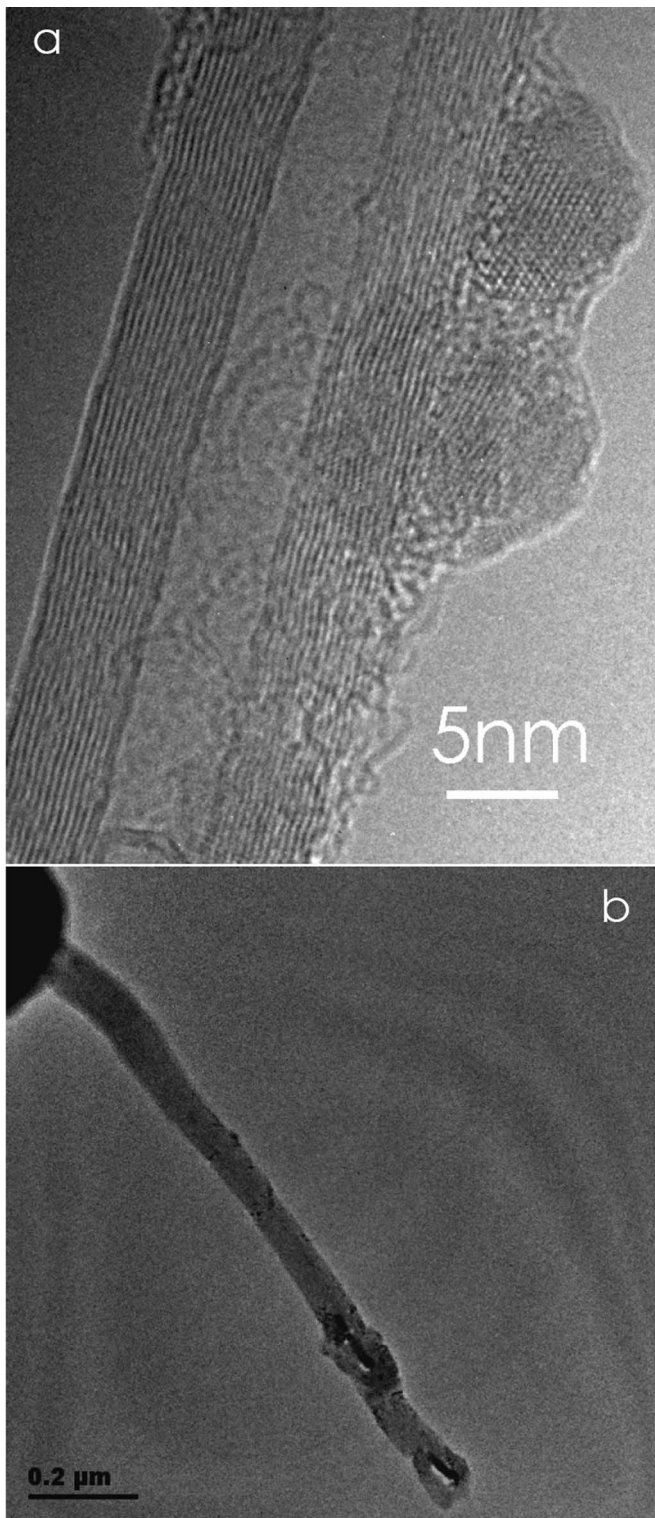


Fig. 4. (a) MWNT with quantum dots (QDs) attachments. (b) Picking up a nanotube with QDs attached using EBID.

shows a high-resolution TEM image of a CNT with QDs attached [28]. Fig. 4(b) shows that a tube is being fixed to a probe after a 30 min exposure to the electron beam (HT: 300 kV), which is typically more than ten times slower than in an SEM.

Compared to EBID, using a “sticky” probe is a more efficient way to attach a tube onto a probe. Fig. 5 shows a Cu-filled

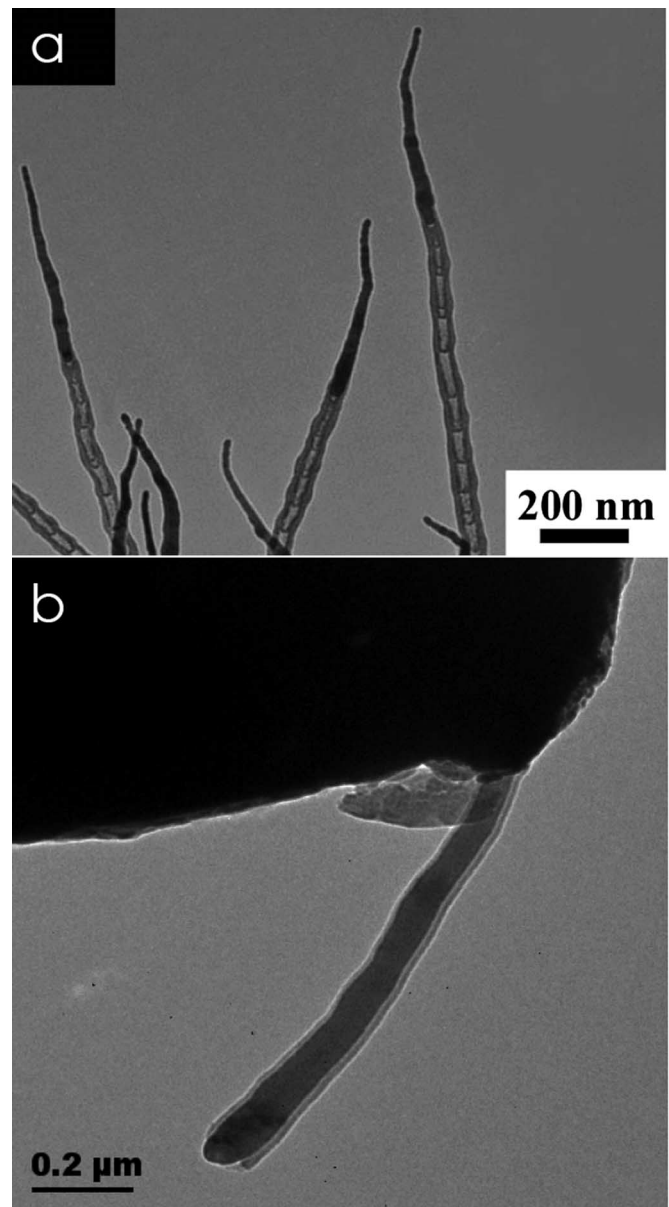


Fig. 5. (a) TEM image of Cu-filled CNTs. (b) Cu-filled nanotube is fixed onto a “sticky” probe and picked up by mechanical pulling.

nanotube fixed to a “sticky” probe and picked up by mechanical pulling. However, it has been found that in the environment of a TEM, the “sticky” probe loses its stickiness very quickly, which might be caused by the high-energy electron-beam bombardment and/or the high vacuum. Moreover, EBID is irreversible and “sticky” probes cannot be reused. New approaches are needed if the purpose is to handle or assemble the tube onto other places rather than fix it onto the probe. Micro-grippers are promising tools for reversible handling, as has been shown inside SEMs [29], but the design of grippers remains a challenge due to the narrow space inside a TEM holder.

The attachment of QDs onto CNTs will create the possibility of assessing the position and orientation of individual decorated CNTs in an aqueous environment as well as the tracking of motion (e.g., for drug delivery). The hollow structures of CNTs

can serve as containers, conduits, pipettes, and coaxial cables for storing mass and charge, or for transport. Because Cu is a good conductor of heat and electricity, and has a very low binding energy when bound to carbon, encapsulated Cu inside nanotubes is ideal for many potential applications. We have investigated the controlled melting and flowing of single-crystalline Cu from CNTs assisted by nanorobotic manipulation; attogram-scale precise delivery of mass has been realized and applications in nanorobotic spot welding have been demonstrated [30].

B. Electric Breakdown for Picking Up

Attachment and subsequent picking up using van der Waals forces is reversible; hence, multiple attempts are possible with the same probe. However, van der Waals interactions between the probe and the tube are not necessarily sufficient for picking up a nonfreestanding nanotube. Here, we show how electric breakdown can help the process. Fig. 6(a) shows a section of a bamboo-structured CNT in contact with a probe while a 10-V bias is applied to probe and substrate (not shown). For a uniform tube, the breaking site can be at any position along the tube. The selection of a breaking point can be realized by preparing an artificial defect such as a kink on the tube. A kink can be introduced by buckling and/or pushing the tube against a substrate [11]. Fig. 6(b) shows a pre-kinked tube being broken at the kink site by the application of a 10-V bias.

Fig. 7 shows the same procedure on a nanotube with QDs attached to it. A bias voltage is applied to the tube starting from 0 mV with a step size of 200 mV. No obvious changes of either the structure or the electric properties of the tube occurred until the bias voltage of 2500 mV was reached. A typical I - V curve is shown in Fig. 7(d). At 2500 mV, the current reaches up to ca. 1 mA, and the tube starts breaking down at the kink site into two sharpened remaining tubes. Fig. 7(d) shows the change of the current and the voltage as the current approaches the saturated value. In the first 6.25 s, the voltage is increased from 2400 to 2500 mV, and the current is increased from 926 to 937 μ A (3.25 s) and then started decreasing. A sharp drop of the current occurred until it reached a stable value around 374 μ A, and the tube peeled accordingly. At 309.75 s, the tube broke completely and the current dropped to zero.

C. Length Control

Length control of a nanotube is significant for many applications. Besides controlled breakdown, field emission current can be used to shorten a nanotube further to a desired length. Fig. 8 shows a nanotube being shortened by using it as a cathode and approaching a probe with a 140-V bias to it, resulting in an oversaturated field emission current.

IV. SHELL ENGINEERING OF MWNTS USING ELECTRIC BREAKDOWN

Fig. 9(a) and (b) shows the peeling of a single layer of an MWNT. The relevant I - V properties have been measured, as depicted in Fig. 10(a) and (b). Due to the exposure of an inner layer, the properties of the MWNT have been obviously

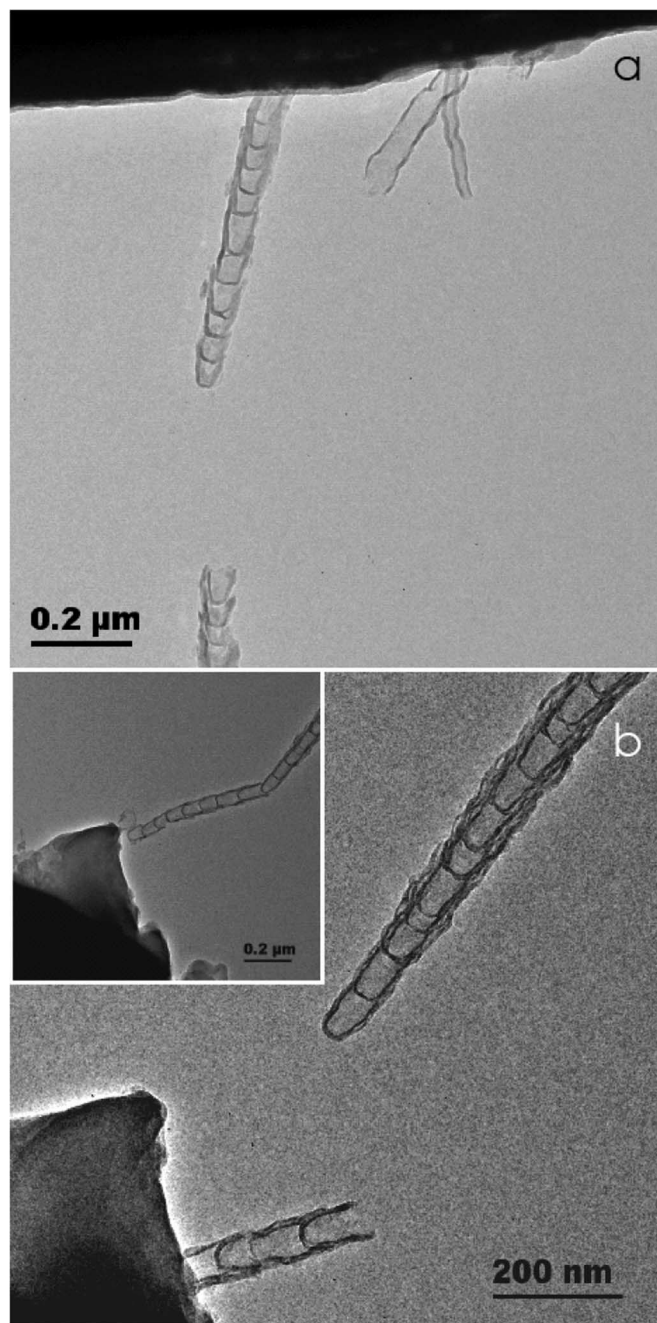


Fig. 6. Picking up a bamboo-structured nanotube using van der Waals force in combination with the electric breakdown (a) a uniformed bamboo-structured tube and (b) a pre-kinked tube (inset shows the preparation of a kink by pushing the tube against the probe before moving it to the final position and performing electric breakdown).

changed. At 1-V bias, the resistance of the tube is 21.89 k Ω , while it was originally 31.09 k Ω before being peeled. So, under this bias, the coupling resistance is 9.2 k Ω . More layers can be peeled by applying higher voltages between the two ends of the nanotube. The resulting tubes are shown in Fig. 9(c) and (d). The current versus time changes were monitored with a multimeter, as shown in Fig. 10(c) and (d). Telescoping motion has been observed in the MWNTs fabricated in this way (Fig. 11).

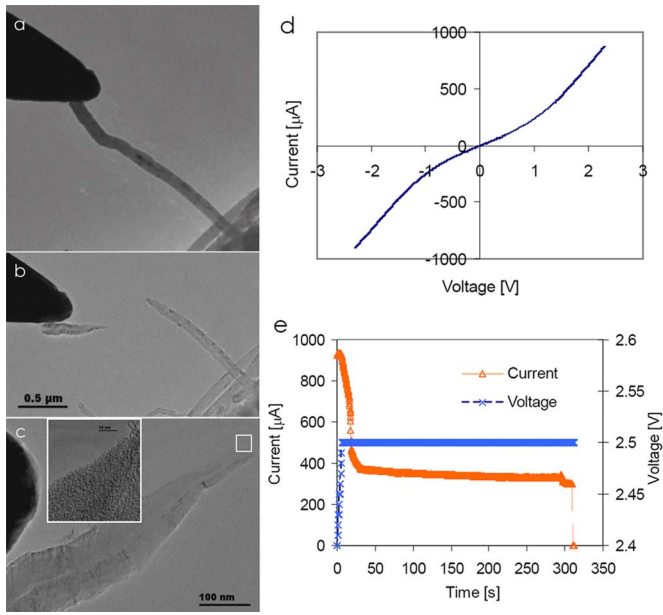


Fig. 7. Picking up a nanotube covered with QDs using van der Waals force in combination with the electric breakdown (a) before and (b) after breakdown. (c) Sharpened tip (inset is a high-resolution image of the blocked part near the end of the tip). (d) I - V curve. (e) I - t and V - t curves.

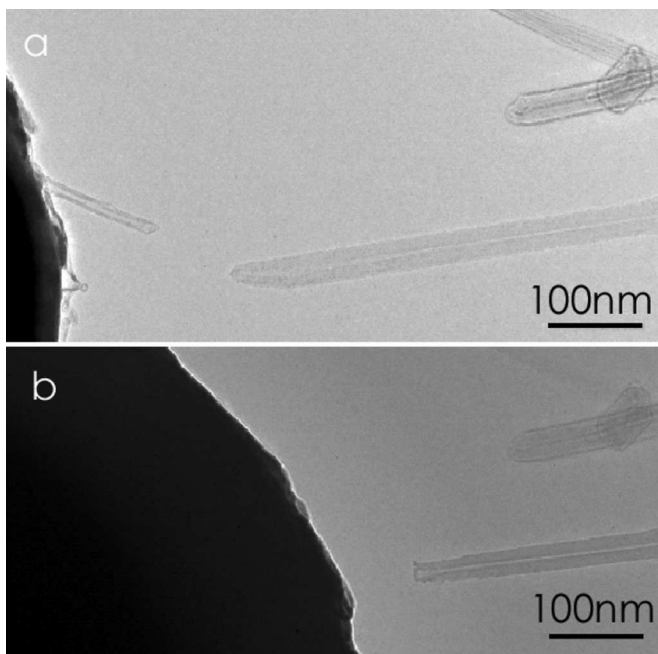


Fig. 8. Shortening/opening a cathode nanotube with oversaturated field emission current by approaching it with a probe. (a) Initial position. (b) End position.

Fig. 12 shows the procedure of electric breakdown of the innermost layer of a section nanotube. A 2.2-V bias voltage is applied to the tube. The changes of the structure and the electric properties of the tube have been monitored using TEM images and a multimeter simultaneously. At certain positions, I - V curves were obtained while keeping the bias voltage constant. As shown in Fig. 12(a)-(f), the tube thinned over time until it broke in the middle. Thermal induced expansion caused

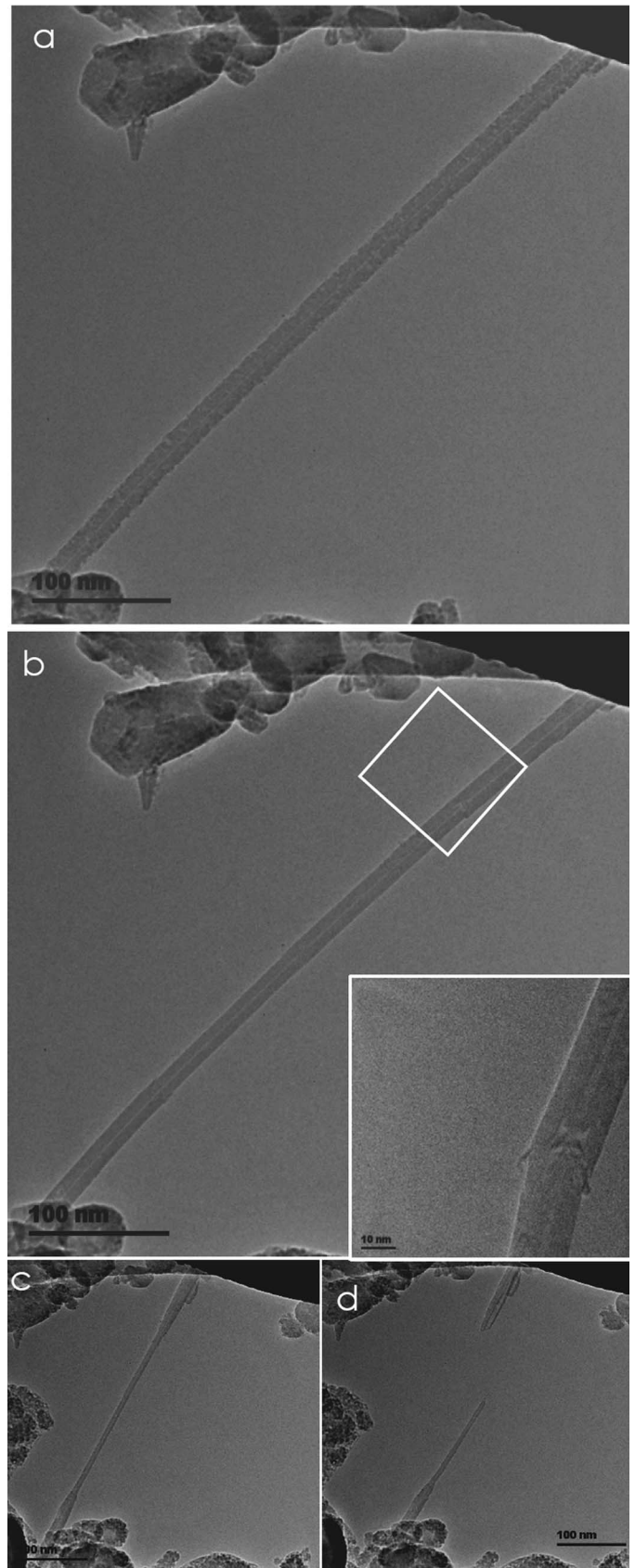


Fig. 9. Controlled peeling of an MWNT. (a) Original tube. (b) One layer (see inset) is peeled under a 2-V bias. (c) Multiple layers are peeled under a 2.4-V bias. (d) Tube is broken under a 2.5-V bias.

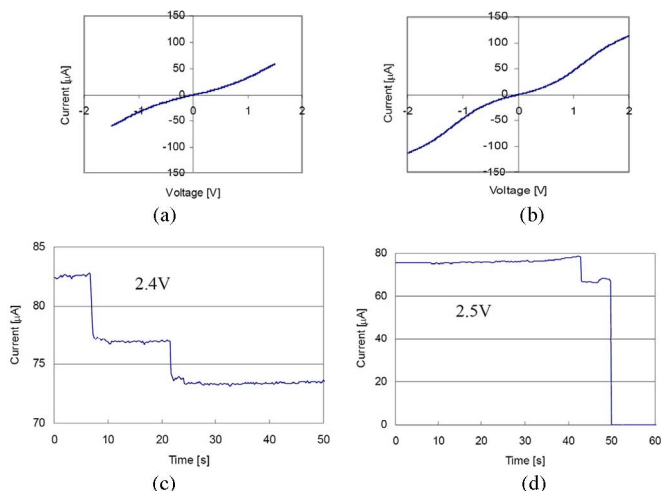


Fig. 10. $I-V$ and $I-t$ curves for controlled peeling of an MWNT. It can be seen from (a) and (b) that the $I-V$ curve changed shape after one layer is broken. The curvature may reflect changes in the interlayer coupling. The plots (c) and (d) show the change of current as the nanotubes peeled as shown in Fig. 9(b) to (c), and (c) to (d), respectively.

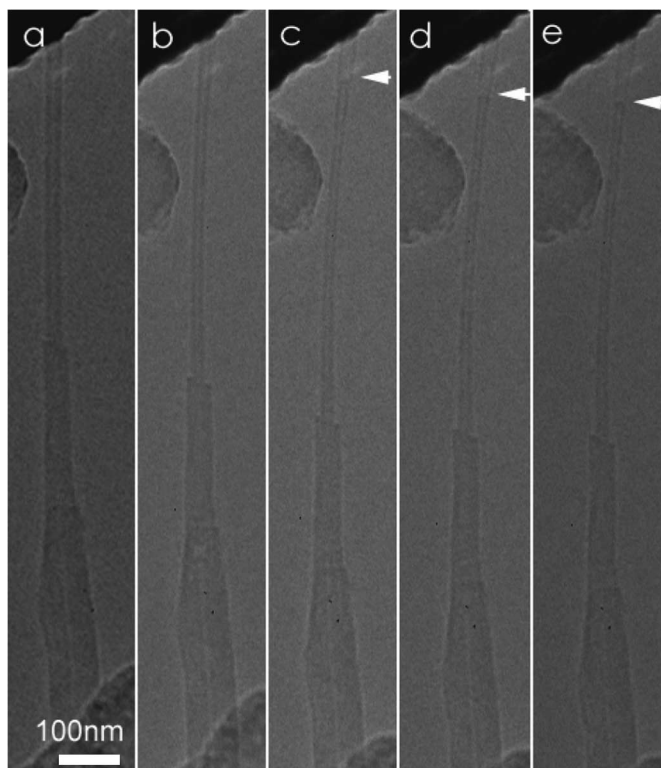
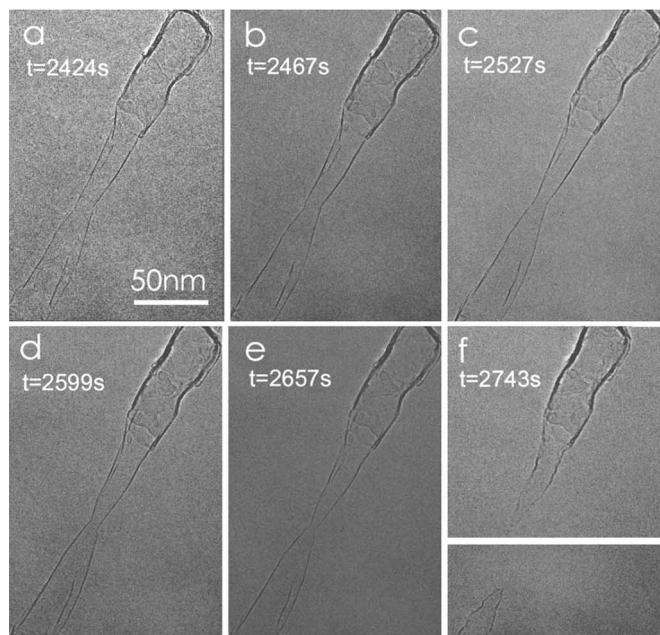
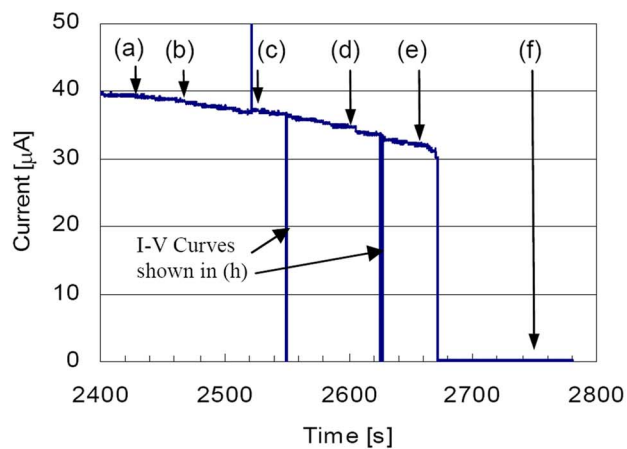
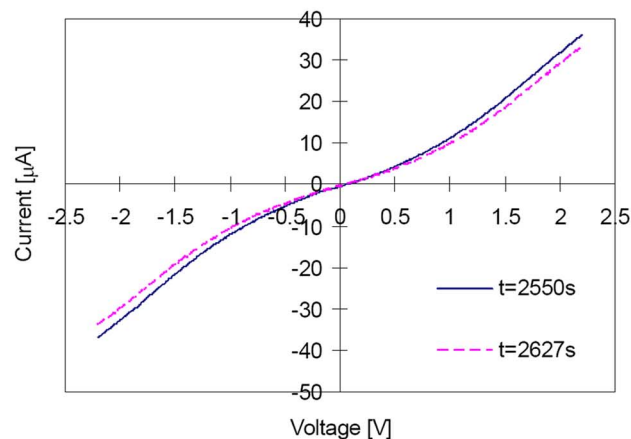


Fig. 11. Telescoping motion realized from electric breakdown MWNTs. The arrow shows the position of the end of nanotubes cores.

the tube to expand. The longitudinal extension makes the tube buckle and an obvious kink was formed as shown in Fig. 12(d). After this, the tube started to break. Fig. 12(f) shows that it broke completely, leaving two sharpened sections on the probe tip and the sample holder. A multimeter recorded the continuous drop of the current [see Fig. 12(g)]. The drop [see also Fig. 12(h)] can be attributed to morphology changes.



(g)



(h)

Fig. 12. Thermal-induced thinning of an innermost layer of an MWNT. (a)–(f) TEM images showing the process of thinning and breaking. (g) $I-t$ curve under a constant bias 2.2 V. (h) $I-V$ curves at 2550 and 2627 s.

V. SUMMARY

Basic processes for nanorobotic manipulation of CNTs inside a TEM have been investigated experimentally including picking up tubes with van der Waals forces, EBID, and “sticky” probes, and electric-breakdown-assisted picking up and length control. MWNTs, bamboo-structured CNTs, CNTs with QDs attached, and Cu-filled CNTs have been used as sample materials. We have demonstrated the suitability of current-driven breakdown of individual nanotube shells for the fabrication of MWNT-based nanoscale bearings. Investigation inside a TEM has correlated the changes in MWNT structures with electrical measurements. Peeling of a single layer allowed the investigation of the interlayer coupling of an MWNT. Electric breakdown of the innermost layer resembles a single-walled nanotube, and has shown a new mode in morphology change, i.e., thinning. These methods are crucial for the characterization of nanoscale materials, structuring of nanosized building blocks, and prototyping of NEMS. Nanorobotic manipulation inside a TEM is enabling both top-down and bottom-up approaches for nanomanufacturing, and stimulating interests for both fundamental research and practical applications. Secondary building blocks from shell engineering can be used as robust nanoscale motion-enabling mechanisms for applications such as bearings, switches, gigahertz oscillators, shuttles, memories, syringes, and actuators. Inner-shell-peeled CNTs can provide larger effective volumes for serving as hydrogen containers, drug capsules, conduits, and pipettes.

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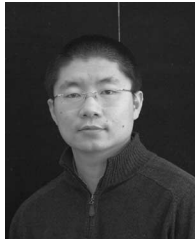


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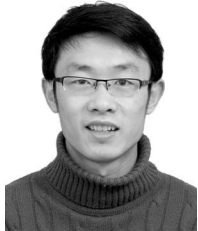


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