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Peeling and Cutting a Multi-Walled Carbon Nanotube inside a Scanning Electron Microscope

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***Abstract*—Carbon nanotube (CNT) has many superior properties if compared to other materials which make it a promising nanostructure to be used in various nanoelectronic and mechanical systems (NEMS). The main problems involved in nanotube based applications are related to the fabrication process and especially to the machining and assembly of CNTs. In this paper, the techniques for peeling and cutting of multi-walled carbon nanotube (MWNT) are presented. Both the techniques take place inside the same observation devise a scanning electron microscope (SEM), which make the combination of the techniques effective. Also the size of the specimen chamber of a SEM enables the use of different fabrication methods like the assistance of a nanorobotic manipulator in different nanomanufacturing steps. The usability of the proposed techniques is verified in the manufacturing of a concept of one dimensional nanoactuator, which can be fabricated by the presented techniques.**

Keywords-carbon nanotubes; cutting; peeling; nanorobotic manipulator; nanodevices

I. Introduction

Nanoscale and especially nanostructures and devices have a great potential to become the next break through of technology. After the discovery of CNT nanotechnology has got a versatile building block with a massive application potential. Still even nowadays there are only few real applications for CNT and the main reason seems to the manufacturing challenges of CNT based nanodevices. A great deal of research has been done to solve these manufacturing problems and several techniques has been developed for CNT machining and manipulation. In several cases, however, the different manufacturing techniques are conducted inside different observation devices which make the combination of the techniques difficult, time consuming and ineffective. Therefore, the efficiency of nanomanufacturing could be increased by conducting the manufacturing steps and techniques inside one observation device. At the same time the reliability of the techniques can be increased because of the decreased sample replacements, in which the sample might get defected.

A precise cutting method of CNT is a prerequisite for the fabrication, manipulation and assembly of nanostructures and systems based on CNTs, because the length of a CNT influences the function and properties of the nanostructure. Another fundamental technique for the manufacturing of NEMS is a precise peeling of a MWNT, which enables the production of nanobearings as the nested tubes are released to slide over the almost optimal sell surfaces [1, 2]. These nanobearings can be exploited in linear or rotating nanoactuators where their offer almost frictionless movements.

A number of methods for the cutting and peeling of MWNT have been investigated previously [3-8]. The most promising of these is the cutting and removal of CNT shells by using a high-energy electron beam within a transmission electron microscope (TEM) [4, 5]. The small size of the specimen chamber of TEM, however, makes it difficult to use the method for nanofabrication processes and for the assembly of nanodevices [9].

In this paper, techniques for the peeling and cutting of MWNT inside a SEM are presented. Because both of the techniques can be conducted inside a SEM, the combination of the techniques is easy, which increases the efficiency of the techniques and makes them very suitable for nanodevice manufacturing. Also the size of the specimen chamber of a SEM enables the use of nanorobotic manipulator in the nanofabrication and assembly as discussed in the paper.

II. Cutting

The proposed cutting technique is depicted in Fig. 1. In the cutting, oxygen is introduced in the vicinity of a CNT and the CNT is exposed on an electron beam by using a spot mode of a SEM. The acceleration voltage of the beam is low which enables the electrons to interact with the carbon atoms of the CNT and makes them active. The active carbon atoms react with the oxygen gas according to the following reactions

$$O_2 + C \rightarrow CO_2 \tag{1}$$

$$O_2 + 2C \rightarrow 2CO \tag{2}$$

In these reactions the carbon atoms react with oxygen atom and forms carbon dioxide and carbon monoxide. The reaction energy is provided by low energy electron beam. It is noteworthy; that the cutting does not happen if either oxygen or electron beam is not involved. The necessity of the electron beam enables accurate positioning of the cutting point, because the cutting happens accurately in the point where the beam is located. The accuracy of the technique makes it a very useful to

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be used in nanomanufacturing applications. The method is also very effective and a single CNT can be cut rapidly. The accuracy and efficiency of the cutting method is tested in an experiment discussed in Chapter IV.

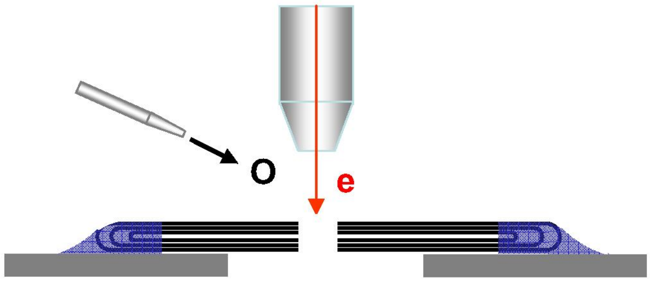

Fig. 1. The procedure of cutting a single CNT inside a SEM by using an electron beam assisted with oxygen gas.

III. Peeling

The proposed peeling technique is closely related to the cutting technique, because also in this technique oxygen is introduced in the vicinity of the MWNT and the activation of carbon atoms happens by using electron beam. Therefore, the peeling happens through the same reactions (1) and (2) as the cutting.

Instead of the spot mode of the electron beam used in cutting, in the peeling a scanning mode is used. The "burning" of the CNT is restricted only on the outermost shell because the oxygen is on the surface of the CNT and can react only with the outermost carbon atoms, not the inner ones. The peeling area can be accurately set according to the scanning area which makes the technique suitable for nanomanufacturing. If compared to cutting, the density of oxygen is normally set lower to prevent the inner layers injure and therefore the peeling takes more time than simple CNT cutting.

IV. Experiment

The proposed peeling and cutting techniques were tested by an experiment conducted with MWNTs with 20~50 nm diameter, synthesized by using the standard arc-discharge method. The experiments were conducted inside a field emission SEM (FESEM, JEOL JSM-6500F). Oxygen gas with a purity of 99.99995 % was introduced into the vicinity of the sample through a glass nozzle with a 20 μm opening at its end. The oxygen gas flow was regulated by a digital mass flow controller. The experiments were assisted with a nanorobotic manipulator constructed inside the chamber.

A. Cutting

In the proposed cutting technique the electron beam with an acceleration voltage of 2 kV and a beam current 3×10^{-10} A is pointed into the desired cutting point by using the spot mode of the SEM. The electron beam was assisted with oxygen gas flow of 1 sccm, flowing from a nozzle sited 500 μm from the CNT.

As a result a single CNT was cut by the electron beam in about 2 min at the left two points and 3 min at the right point as can be seen from Fig. 2. Figure 2(a) shows the CNT before cutting, Fig. 2(b) shows the CNT after cut at three points. The cut off length are 70 nm, 45 nm and 165 nm, respectively.

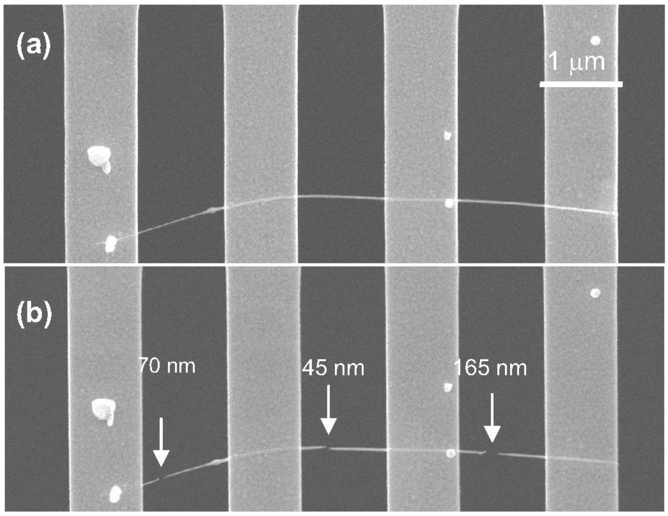

Fig. 2. (a) Before and (b-d) after cutting of a single CNT. (b) The cut off length are 70 nm, 45 nm and 165 nm, respectively.

As shown by the experiment, the proposed cutting technique is accurate, effective and relatively easy to conduct inside a SEM. The cutting speed can be controlled by controlling the acceleration voltage, irradiation current or oxygen density. It was also found during the experiment, that if the gas nozzle is too far from the sample, i.e. the oxygen density is too low, no cutting occurs. The cutting happens neither, if the acceleration voltage of the electron beam is too high. Therefore the essential conditions for the cutting are the sufficient oxygen density on the surface of the CNT and a low energy electrode beam. Experiment results have been reported in ref. [9].

B. Peeling

The proposed peeling technique was tested by using the same experiment arrangements as in the cutting. During the experiment, the electron beam was set to a scan mode with a scan area of 0.8 × 0.2 μm. The acceleration voltage and the irradiation current were 2 kV and 3×10^{-10} A, respectively, and the oxygen gas flow was 1 sccm.

The scanning area was exposed to the electron beam for 30 minutes and the results are shown in Fig. 3. The peeled MWNT with the black box indicating the scanned area is shown in the upper figure and the magnification of the area of the white box in the lower figure. As can be seen, the diameter of the MWNT is smaller in the area exposed to the electron beam. This is because the outer shells of the MWNT are etched by the electron beam.

Because the peeling of a CNT can be done by using the same kind of experiment device as the cutting, these two techniques can be easily combined. This makes the techniques very suitable for nanodevice manufacturing as will be discussed next.

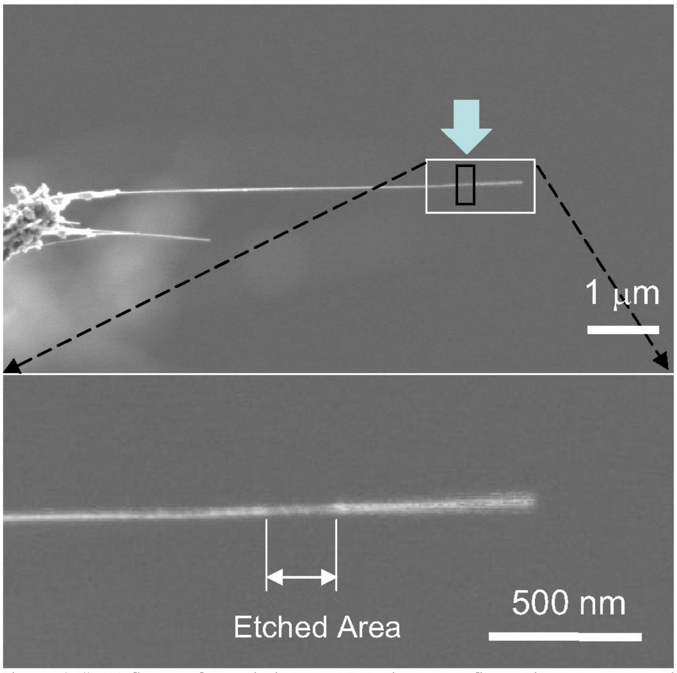

Fig. 3. A SEM figure of a peeled MWNT. In the upper figure the area, exposed to electron beam, is marked as black square. In the lower figure a magnification of the upper figure is shown, revealing the absence of the outer shells.

V. Applications

A CNT is a promising nanostructure to be used in various nano-electronic and mechanical systems. One interesting such a system is a linear actuator where the almost perfect sliding surfaces of a MWNT are used as linear bearings of the actuator. The actuator can be constructed from a single MWNT by using the nanomanufacturing techniques discussed earlier.

The manufacturing steps of the actuator are presented in Fig. 4. First a MWNT is placed on a holder on its center (Fig 4 a). This can be done by using a nanorobotic manipulator. After that the length of the MWNT is adjusted to be appropriate by the proposed cutting technique (Fig 4 b). The outer ends of the MWNT can then be removed by using the proposed peeling technique. The technique should not harm the inner layers of the CNT and therefore the inner shells should be free to slide inside the optimal bearing formed by the outer shells (Fig 4 c). The inner shells can be now moved by placing electrodes close to the ends of the tube and introducing a voltage over one of the tube ends and electrodes (Fig. 4 d). The direction of the movement can be changed by changing the end which the introduced voltage affects. Also the movement can be controlled by controlling the voltage balance of the tube ends.

In the actuator design the electrical force affecting in the tube ends can be calculated according to

$$F_{Ci} = \frac{1}{2} U^2 \frac{d}{dz} C_g \tag{3}$$

where

U = Voltage between the electrode and tube end [V]

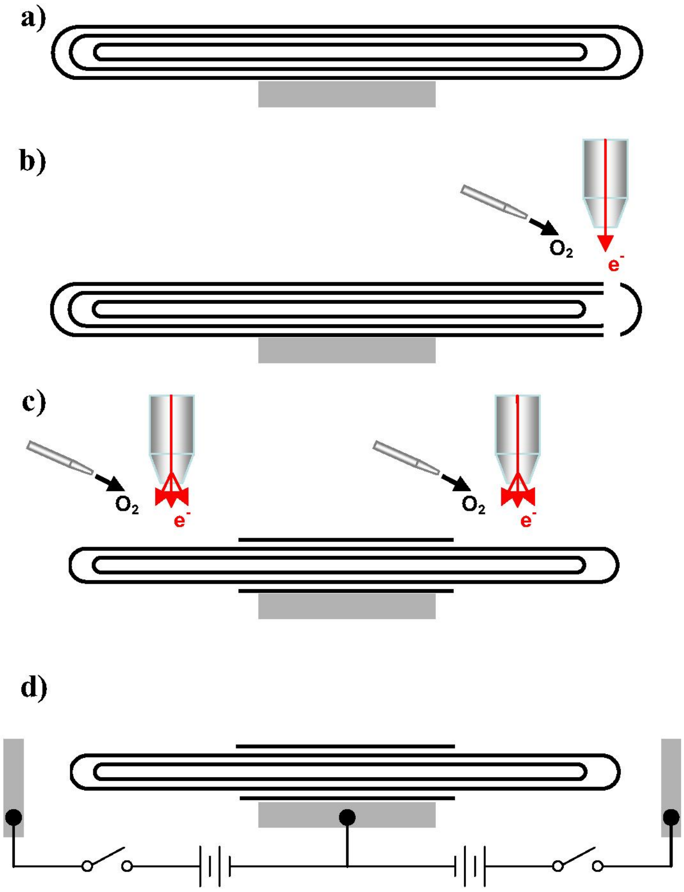

Fig. 4. The construction process of the proposed linear actuator concept produced by using the presented techniques from a single MWNT. a) A MWNT is placed on an substrate by using a nanorobotic manipulator. b) The length of the MWNT can be set accurately by using the proposed cutting technique. c) The ends of the outer shells are removed by using the proposed peeling technique. d) The inner shells can be moved by an electrical force induced by the voltage between the CNT and electrodes.

z = Distance between the electrode and tube end [m]

C_g= Capacitance between the electrode and tube end [F]

The capacitance can be approximated by the

$$C_g = \frac{4\pi\varepsilon_0 r}{\ln\left(\frac{2z}{r}\right)} \tag{4}$$

where

ε_0 = permittivity of vacuum (~ 8,854187 x 10^{-12} [F/m])

r = radius of the inner tube [m].

By calculating the derivative dC_g/dz and applying it into the equation (3) we get:

$$F_{Ci} = \frac{\pi\varepsilon_0 L}{\left(\ln\left(\frac{2z}{r}\right)\right)^2 z} U^2 \tag{5}$$

Because, neither ends of the inner tube are inserted into the outer shell, the inserted area is constant during the movement. In that case, there is no effective van der Waals force resisting the movement and its effect can be neglected [10]. Note that the case is different if the movement is so long that one of the ends is inserted into the outer shell. In that case there is a constant effective van der Waals force and its strength can be calculated from Lennard-Jones Equation [10].

Although, the nested CNTs form an almost optimal sliding surface, there exist microscopic mechanisms against sliding [11]. There seems not to be a consensus about the mechanisms of the sliding resistant, but there exist two theories about the mechanism. The other assumes that the sliding resistance is proportional into the nested area while the other assumes that the mechanical resistance is more or less a point formed force and is therefore independent from the nested area [12, 13].

An experiment that the inner layer was slide on the outer shells driven by a nanorobotic manipulator has performed as shown in Figs. 5 and 6. A single MWNT was suspend on two AFM cantilever, one end of MWNT was fixed on cantilever surface using electron-beam-induced deposition and another end was attached with van der Waals force. A part of outer layer at the bottom of MWNT in Fig. 5(a) was removed using the peeling technique. The exposed inner shells are 260 nm in length. Figures 5(b)-(d) show that the inner shells were drawn from the outer shells with the nanomanipulator. The inner layer was gone back as shown in Figs. 6(a)-(d).

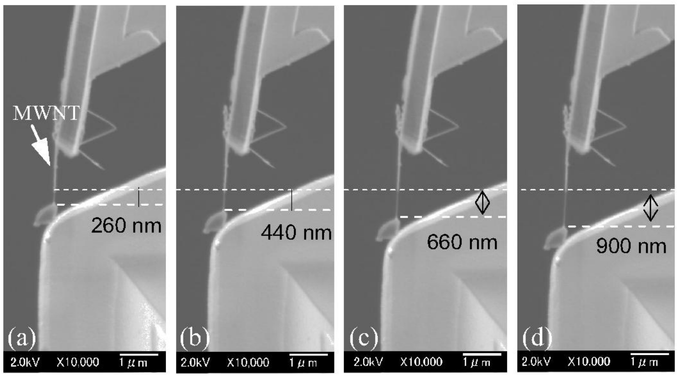

Fig. 5. The inner layer was drawn from the outer layer by the nanomanipulator.

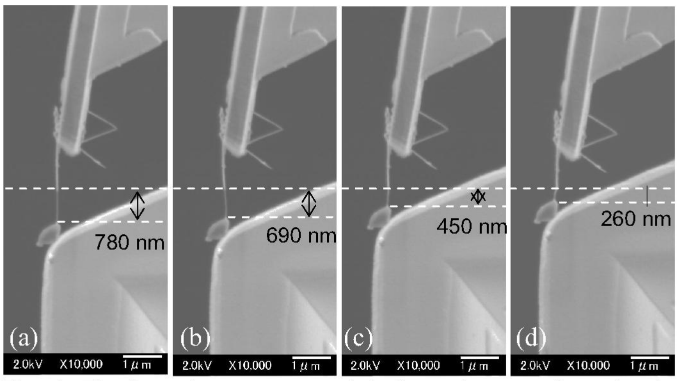

Fig. 6. The inner layer was moved back to the outer layer by the nanomanipulator.

VI. Summary

Two fundamental nanomanufacturing techniques, precise cutting and peeling methods of a CNT, were presented in this paper. The techniques were tested and the experimental results indicate that the CNT can be precisely cut inside a SEM by the electron beam based cutting technique, assisted with oxygen gas. The cutting of CNT by the technique is precise and rapid. It has also been shown, that the outer shells of MWNT can be precisely etched by the proposed technique.

Because the techniques can be conducted inside a SEM with a size of a relative spacious specimen chamber, the techniques enable effective nanofabrication processes and nanoassembly based on CNTs. It has been demonstrated through the sliding of inner shells driven by the nanomanipulator that the nanofabrication methods can make a moveable structure from MWNTs. In the next work, we will use those techniques to manufacture a one dimensional nanoactuator.

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