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Nanorobotic Manipulator Assisted Assembly of Complex Nanostructures based on Carbon Nanotubes

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Abstract— **In this paper, a series of *in situ* nanofabrication techniques of nanostructures, including cutting, bending and welding of carbon nanotubes (CNTs), inside a field emission scanning electron microscope (FE-SEM) is reported. In the CNT cutting technique, a CNT was exposed to a low energy beam assisted with 1 sccm oxygen gas flow. The proposed technique is effective being capable of cutting a CNT in less than 1 minute. It was found that, although the total pressure of specimen chamber reached 10^{-2} Pa, high speed cutting occurs only in the area close to the nozzle. The presence of oxygen gas in the vicinity of the CNT can be applied also for the bending of CNT, if some conditions of the cutting technique are changed. These include the increase of the acceleration voltage and/or setting the oxygen gas nozzle farther from the sample, and/or reducing the irradiation time. Using the proposed bending method the angles larger than 90° can be formed and the location of the kink can be set accurately. It is also shown, that tungsten can be deposited on a substrate by the electron-beam-induced deposition (EBID), if the oxygen of the proposed cutting technique is replaced by $W(CO)_6$. In this paper, these three nanofabrication methods were exploited in creation of a two dimensional (2D) nanostructure, the letters N and U, and a three dimensional (3D) nanostructure, the letter N. The 2D letters were constructed from 6 CNTs assembled on a substrate while the 3D letter N was bended from a single CNT and fixed to stand on a substrate. Based on the high performance of the proposed techniques, it is suggested that the cutting, bending, and welding techniques inside SEM will become widely utilized in the fabrication and assembly of nanodevices and in the characterization of nanomaterials.**

I. INTRODUCTION

In recent years, carbon nanotubes (CNTs) have been proposed as a basic building block for a new generation of nanoelectronic and mechanical systems. Previous researches showed that CNTs can be used as linear [1] and rotational nanobearings [2], mass conveyors [3], field emitters [4, 5], atomic force microscope (AFM) probes [6], nanotweezers [7], nanoposition sensors [8] and so on. All of those applications are based on as-grown CNTs without any change in the mechanical structure of CNTs except some chemical doping [9]. In fabrication, manipulation or assembly of nanotubes, the length of CNTs is an important factor and will influence the function and structures of the nanostructures and nanodevices. Therefore, a method of defining the length of nanotubes precisely is indispensable. The applications of CNTs in special structure or shapes can be significantly improved by a method capable of making an irreversible bending of CNTs. In addition of these structure manipulation methods, also an effective welding technique is essential for the development of CNT based nanostructures and devices. The reliability and efficiency of all these nanofabrication methods can be improved, if they can be completed inside the same equipment, which decrease the risk of destruction of the structure and electronic properties by minimizing the need for the opening of the vacuum chamber. This would also make the combination of the methods more effective which is needed if more complicated structures are manufactured.

The peeling and sharpening of multi-walled carbon nanotubes (MWNTs) through electrically driven vaporization presents a possible method for the removal of CNTs [10], but this method is difficult to control and needs an electrical contact to the CNT. Cutting or removal of CNTs on a nanometer or even an angstrom scale with high-energy electron beam inside a transmission electron microscope (TEM) was report by F. Banhart *et al.* [11, 12]. Although, by using a TEM, CNTs can be cut at high resolution, the small specimen chamber of TEM make nanofabrication and assembly difficult, which restrict the applicability of the method in the nanodevice fabrication. Also destructive fabrication technique is proposed for the cutting of CNTs, but unfortunately the destructive point is unknown and difficult to control, which makes the method unsuitable for precise nanomanufacturing [13]. The other mechanical cutting methods, like the cutting with an AFM [14] or a scanning tunneling microscope (STM) tip [15], are time consuming and therefore not very effective for complicated applications. Also the CNT etching via focused ion beam (FIB) is proposed, but the FIB could damage the rest of nanotube, which is undesired in nanofabrication [16].

Suzuki *et al.* reported that the electron beam irradiation induce damages on CNTs under the low acceleration voltage of a scanning electron microscope (SEM). This method was applied in selective removal of nanotube with a beam accompanied with oxygen, as reported in [17]. As another SEM based method, *In situ* cutting CNTs with water vapor inside an environmental SEM has reported by Zettl et al.

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They showed mass loss of CNTs caused by water molecular [18]. Despite the variety of the cutting methods, none of these researches have explained why the CNTs are cut under low-energy electron beam. As we know, for removing a carbon atom by a knock-on collision, minimum incident electron energy of 86 keV is required [19]. The high-energy beam can be easily obtained inside a TEM, but in a SEM, the energy of electron beam is normally limited to 30 kV. Therefore, the cutting mechanism inside a SEM has to differ greatly from the one inside a TEM.

Reversible bending of CNTs with AFM was reported by M. R. Ralvo *et. al.* [20]. They found that the carbon nanotube is a material with extraordinary strength through the bending experiments. The AFM method is restricted by the difficult fabrication of the suspended structure. Bending of MWNTs with manipulation inside a TEM was performed for the dynamical measurement of the electrical conductivity of CNT during the deformation [21]. Plastic deformation of double-walled carbon nanotubes was also induced by applying current over the nanotube [22]. The location of the kink is, however, difficult to define in these two methods were manipulation and current are used. They are not capable of sharp angle formation either. W. H. Knechtel reported a reversible bending induced by electron beam inside TEM [23]. The bending was caused by van der Waals force between the CNT suspended on two gold lines and the substrate.

Bending can not only be used for building nanostructures but also for nano electronic devices. The electrical conductivity properties of a single-walled carbon nanotube with an intramolecular junction were measured in [24]. The results show that the metal-semiconductor junction behaves like a rectifying diode with nonlinear transport characteristics. The sharp angles can be made also by using FIB, but the method easily damages the sample, which is not favorable [25].

An effective welding method for fixing a CNT on substrate or connecting two CNTs is desired. One possibility would be the exploitation of van der Waals forces by which, for example, a CNT can be attached on the surface of a probe during the pick up the CNT. The van der Waals force between the CNT and the substrate is, however, always less than the strength of the nanotube and therefore the method is not suitable for applications were high binding strength is needed. The other possibility is electron-beam-induced deposition (EBID) which can be performed inside a SEM as will be discussed.

In this paper, techniques for high speed cutting and bending of CNTs by introducing oxygen gas into the vicinity of the sample are presented. In addition, an effective welding method of CNTs by using electron-beam-induced deposition with $W(CO)_6$ as precursor is reported. Finally, the usefulness of these methods is demonstrated in *in situ* nanofabrication of a two (2D) and three dimensional (3D) nanostructures based on a CNTs.

II. EXPERIMENTS

A nanorobotic manipulation system [26, 27] with 16 degrees-of-freedom (DOFs) was used in three dimensional manipulations of CNTs. The manipulator has been previously used successfully in a control of a single nanowire grown [28]. The manipulator is actuated with Picomotors^{TM} (New Focus Inc.) for coarse motion and PZTs for fine motion. The manipulator was operated inside the FE-SEM (JEOL JSM-6500F). Resolution of the manipulator is greater than 30 nm (linear) and 2 mrad (rotary) for coarse motions and within a nano-order for fine motions.

During the experiments, a bundle of MWNTs were fixed on a stage inside a FE-SEM by electrically conductive tape. The MWNTs typically 20~50 nm in diameter were synthesized by the standard arc-discharge method. An individual MWNT was picked up from the CNT bundle by the nanorobotic manipulator and was fixed on the tip of an AFM cantilever with EBID [29, 30]. Tungsten hexacarbonyl, used as a precursor, was filled into a glass tube with a 0.8 mm diameter at the open end. The gap between the nanotubes and the gas nozzle was controlled also by the manipulator.

Oxygen gas (purity of 99.99995%) was introduced to the vicinity of the sample through a glass nozzle with a 20 μm opening in the end and regulated by a digital mass flow controller. CNTs were observed in an acceleration voltage of 5 kV, cut in 1 kV and bend in 2kV inside the FE-SEM. We selected the point mode of the beam for cutting and bending CNTs. The pressure in the specimen chamber was increased from 10^{-4} to 10^{-2} Pa during introduction of the oxygen gas flow (1 sccm). For getting clear structures about cutting point, TEM images were taken before and after the cutting process in a JEOL 2100 TEM using acceleration voltage of 200 kV.

III. RESULTS AND DISCUSSION

A. Cutting of CNTs

A single CNT was cut by the electron beam assisted with oxygen gas as shown in Fig.1. The cutting was performed under a constant electron beam current (60 nA), acceleration voltage (1 kV), vacuum pressure (1.6×10^{-2} Pa) and oxygen gas flow (1 sccm). The gas nozzle was at 90 μm distance from the CNT. Figure 1(a) shows the CNT before cutting, (b) shows the CNT after cutting 650 nm long at the 1st time, and (c) was cut 700 nm long at the 2nd time. Figure 1(d) shows the cutting result: the CNT was trimmed to have the same length as the left CNT. The experiment result justifies that the length of CNT can be precisely controlled by the oxygen gas assisted electron beam. Different combinations of acceleration voltages and beam currents, which enable CNT cutting in less than 1 minute, where tested and are show in Fig. 2.

Figure 3 shows the nanotubes deposited on a TEM grid before and after cutting. Figure 3(a) shows the nanotube suspended across a gap before cutting. The cutting was performed inside a SEM. Figure 3(b) shows the CNTs after cutting and as can be seen, about 100 nm of material was removed. It can be also detected that the gap is larger than the beam spot size (3~5 nm), and that the tip of CNTs after cutting is sharpened. The main reason for this is the drift of the beam.

Also two other experiments were conducted in the same experiment setting, but with slightly different conditions. In the first experiment, a CNT was irradiated under low-energy electron beam without oxygen gas. In the second experiment, oxygen gas was introduced, but the gas nozzle was far from the sample. The results are shown in the following.

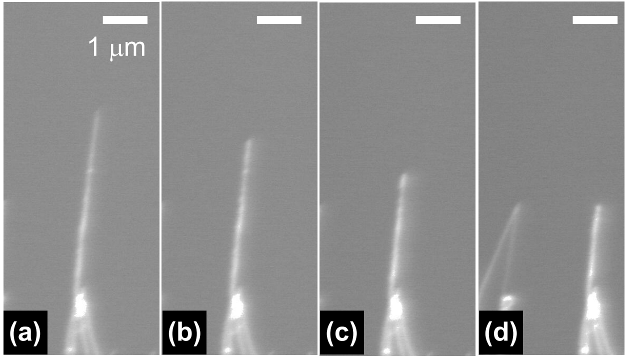

Fig. 1. A single CNT (a) before and (b-d) after cutting. A CNT (b) after 1st cut of 650 nm, (c) 2nd cut of 700nm and (d) finally having the same length as the CNT on the left.

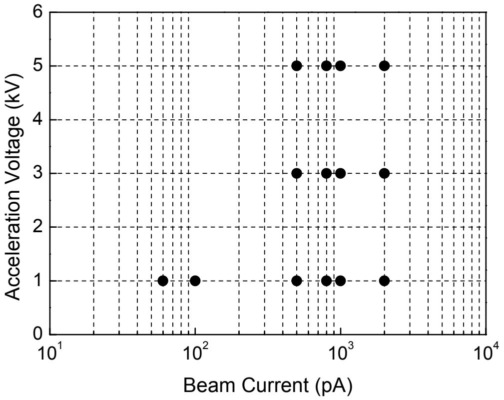

Fig. 2. Cutting CNT less than 1 min under various acceleration voltages and beam currents shown by the black circles.

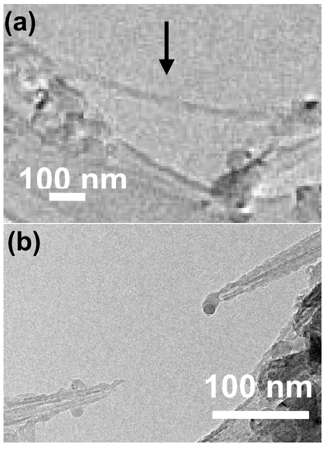

Fig. 3. TEM image of MWNTs before (a) and after (b) cutting, about 100 nm of materials were moved.

1) A single carbon nanotube exposed to the electron beam without introduing oxygen gas

A single CNT was irradiated with an electron beam current of 6×10^{-10} A, an acceleration voltage 1 kV, and without introducing oxygen gas. The vacuum pressure of specimen chamber was 2.7×10^{-4} Pa. The electron beam was set point mode and an area 500 nm away from the tip of CNT was irradiated. The sample was examined after 0, 5, and 10 minutes, but no variation in the CNT was found.

2) A single carbon nanotube exposed to the electron beam with introduced oxygen gas from a distant nozzle

A single CNT was irradiated with the same parameters as in the previous case, except that the oxygen gas of 1 sccm was introduced to the specimen chamber. The gas nozzle was 1 mm from the sample. Because of the gas flow, the total pressure of specimen chamber was 1.6×10^{-2} Pa. The irradiated sample was examined again after 0, 5, and 10 minutes. As a result, the CNT was encountered no variation, except a bending of a 4 degree angle after 10 minutes.

As shown by the experimental results, the cutting of a CNT under SEM needs to fulfill two conditions. The first is the low-energy electron beam giving damage to the CNT, and the second is the oxygen gas reacting with carbon molecules. The details of the cutting mechanism have been reported on previous report [31].

B. Bending of CNTs

The presence of oxygen gas in the vicinity of the CNT can be used also for the bending of CNT. The mechanism of bending is similar to the cutting. This is because the carbon nanotubes can not keep their straight structure, if the carbon-carbon bonds of hexagonal carbon lattice are destructed and part of carbon molecules are removed by the oxygen molecules. Therefore the CNT bends to the direction of the destruction where the carbon molecules form new bonds with each others over the defect. Some theoretical calculations about bending have been reported in [32-35]. The only changes needed in the process conditions are the increase of the acceleration voltage or the receding of the oxygen gas nozzle from sample, or reducing the irradiation time. The bending procedure was demonstrated in the following experiment.

In the experiment, the gas nozzle was setup in 170 μm distance from the sample. The acceleration voltage and the irradiation current were 2 kV and 3×10^{-10} A, respectively, and the oxygen gas flow was 1 sccm. An individual CNT was irradiated in point beam mode for 2 min or 30 s at three points, and the results are shown in Figs. 4. Figure 4(a) shows the carbon nanotube before bending. In Fig. 4(b) a point on the CNT was irradiated 2 min and a clear angle can be observed in the respective point. Irradiation was continued for 30 s on the same point which increased the angle as can be seen from the Fig. 4(c). The experiment was repeated in the other location of the CNT and after 2 min irradiation approximately same angle was bended in the CNT as in the first experiment, as shown in Fig. 4(d). From the Fig. 4(e) it can be seen that the 30 s additional irradiation was able to increase the angle as in the first experiment. After additional 2 min irradiation an over 90 degree angle was formed, as shown in Fig. 4(f).

The experiment clearly shows that the CNT can be bended accurately by using the proposed technique in the means of bending point and the bending angle. The bending process can be executed with the same experiment settings as the cutting.

C. *Welding of CNTs*

Tungsten can be deposited on a substrate by the EBID, if the oxygen of the above system is replaced by $W(CO)_6$. In this technique the EBID is caused by the dissociation of molecules adsorbed to a surface by high energy electrons. The technique was demonstrated in an experiment where three spots were deposited on a surface of an AFM tip by using the technique. The spots were deposited with three different emission currents 0.01, 0.03, 0.10 nA, respectively. The deposition time was 3 min for every spot. The spots are shown in Fig. 5(a) along with the respective diameter values. As can be observed, the higher emission current speeded up the deposition process. The deposition technique can be used in CNT welding i.e. the CNTs can be fixed on a substrate as shown in Fig. 5(b) or connected to other CNTs. This allows a construction of a complicated CNT structures to be used in the nanofabrication of nanodevices. Because the use of the welding technique needs only small changes in the concept used in the cutting and bending techniques, these three techniques can be effectively combined in the nanofabrication applications.

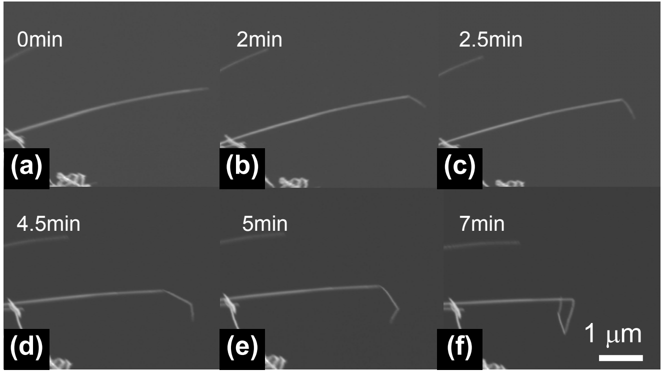

Fig. 4. Bending of a CNT for 2 min or 30 s at three points, respectively.

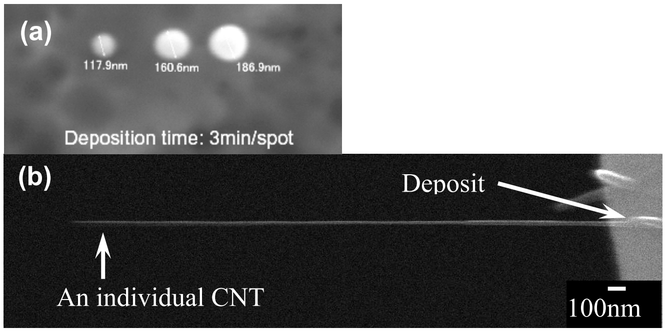

Fig. 5. (a) Three spots are deposited at each 0.01, 0.03, 0.10 nA and with the same 3 minutes deposition time. (b) An individual CNT was fixed on substrate.

As an example of such, two nanostructures were constructed by using these techniques as discussed next.

IV. ASSEMBLY OF CNTS ASSISTED WITH CUTTING, BENDING, AND WELDING PROCESS

A. *Two-dimensional nanostructure*

Because of the great effect of the CNT length to the functionality of the nanostructure, the effective cutting techniques are indispensable for nanofabrication and nanostructure assembly. The usefulness and functionality of the proposed cutting technique is shown in this experiment where a nanostructure is created from precisely cut CNTs, cut by using the cutting technique and fixed by using the welding technique. Fig. 6(a) shows an array of eight tungsten dots deposited with FIB on a thin gold film coated substrate. The dots are 1 µm in diameter, 350 nm in height and have a 1.5 µm intervals. The side view of the dots is shown in Fig. 6(b). The nanostructure was created by picking up a single CNT with an AFM cantilever and suspending it across two dots by using a nanorobotic manipulator as shown in Fig. 6(c). The other end of a nanotube was then fixed by using EBID on the top of the dot and the nanotube was strained so that the tension was stronger than the adhesion between the CNT and the substrate. After that also the other end was welded and the excess nanotube was cut by using the cutting technique described in previous sections. The resulting stretched CNT is shown in Fig. 6(d). The cutting point is indicated by the arrow in the figure. After that the rest CNT were assembled by applying the same procedure. The final result was the letters N and U being assembled with 6 CNTs based on eight tungsten dots as shown in Figs. 7.

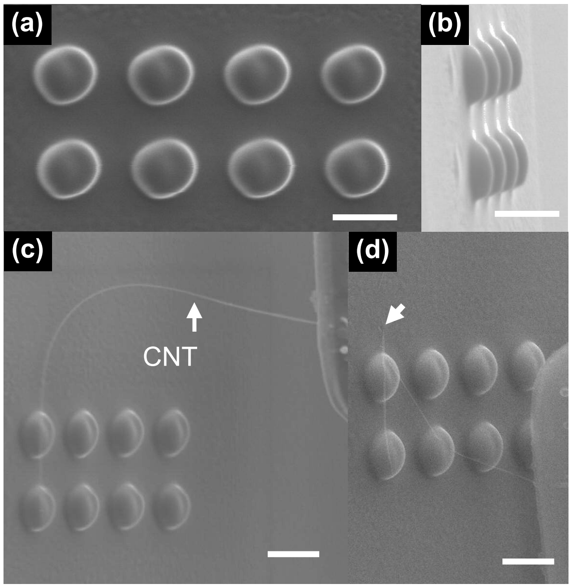

Fig. 6. Top view (a) and side view (b) of the tungsten dots deposit with FIB on gold coated substrate, a carbon nanotube picked up by an AFM cantilever and set on the tungsten dots (c), cutting process for separating CNT from substrate, scale bars are 1 µm.

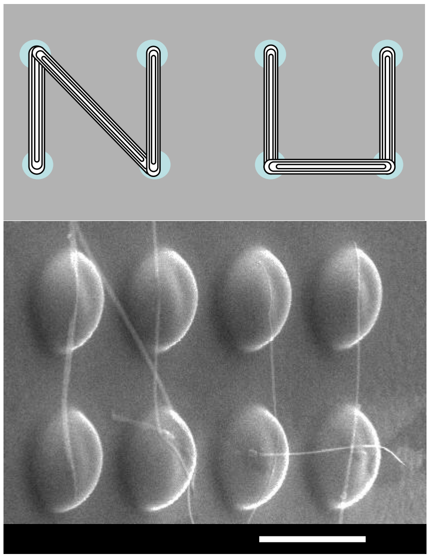

Fig. 7. Letters NU were assembled with 6 carbon nanotubes. Bar is 1μm.

B. Three-dimensional nanostructure

The combination of the welding, bending and cutting techniques and the nanorobotic manipulation is demostrated in an experiment where a 3D nanostructure is constructed. The assembly progress of the structure is shown step by step in Figs. 8. Figure 8(a) shows a CNT picked up by an AFM cantilever and manipulated by the nanorobotic manipulator. The other end of the CNT was fixed on AFM cantilever surface by a tungsten deposit, produced by the proposed welding technique. The other end was set to touch the surface of another AFM cantilever. The proposed bending technique was applied on the CNT and, as can be seen from Fig. 8(b), the CNT was bent at this point. The direction and angle of bending can be controlled by the manipulator.

The first bending was followed by another bend in other CNT point, as shown in Fig. 8(c). The location and orientation of the CNT was changed by the manipulator and the second knick was set to touch the substrate as shown in Figure 8(d). Finally, the CNT was cut at third point shown in Figure 8(e) and the created 3D nanostructure was separated from the substrate one.

As the result, a letter N was assembled in a CNT and stand on the substrate at two points as shown in Figs 9. The two points attach the structure on the substrate only by van der Waals force.

V. CONCLUSION

A series of *in situ* nanofabrication technique of CNTs including cutting, bending and welding inside FE-SEM were reported. In the cutting technique of CNTs a low-energy electron beam is assisted with oxygen gas and as a result a CNT was cut in less than 1 minute. The induced oxygen gas was regulated by a digital mass flow controller and a 1 sccm oxygen flow was injected from a glass nozzle located in the vicinity of the CNT. In the experiment it is found that, although the total pressure of specimen chamber reached 10^{-2}Pa, the high speed cutting occurs only in the area close to the gas nozzle. The same technique, i.e., the electron beam and the oxygen gas in the vicinity of the CNT, can be used also for the bending of CNT. However, in the case of bending, some conditions of the system have to be changed from those of the cutting. These include the increasing of the acceleration voltage and/or disentangling of the oxygen gas nozzle from the sample, and/or reducing of the irradiation time. The bending method was shown to be capable of creating a shape angle in precise set locations. The proposed cutting technique can be applied also in a deposition of tungsten on a substrate, if the oxygen of the cutting concept is replaced by $W(CO)_6$. This method, called as EBID, can be used as a welding of CNTs on the substrate or on another CNT.

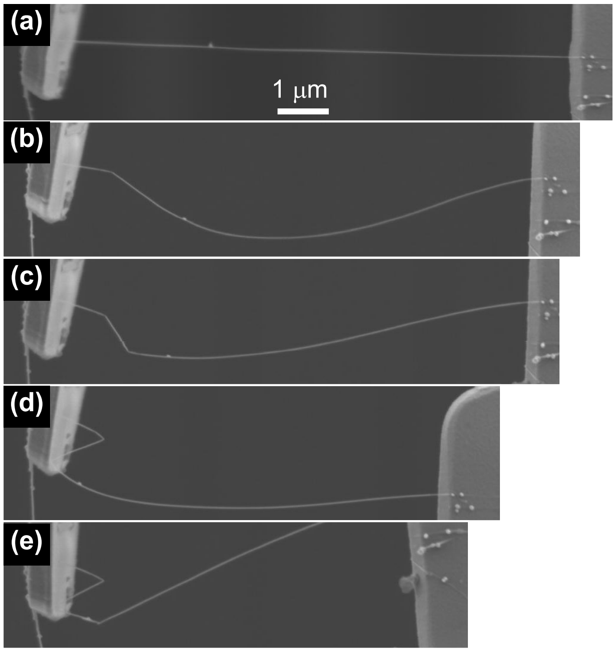

Fig. 8. Assembly of three dimensional nanostructure based on a CNT assisted with welding (a), bending (b)-(d), and cutting (e) techniques.

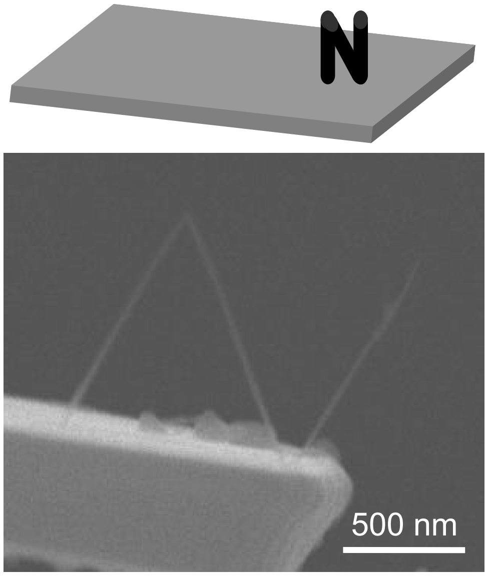

Fig. 9. A letter N assembled with a single CNT stand on a substrate.

Because the proposed nanofabrication techniques can be conducted inside one specimen chamber, the specimen chamber of a SEM, and by using a similar experiment concept with only a slightly changes, they can be effectively combined in different nanomanufacturing processes.

As examples of such a manufacturing process, two nanostructures were created by using these three nanofabrication techniques. The first structure was a 2D nanostructure consisting of two letters N and U. The letters were assembled from a 6 CNTs which were strained over EBID dots. The other structure was a 3D nanostructure, the letter N, bended from a single CNT and set to stand on a surface of a substrate. These experiments show the efficiency of the proposed techniques and it is assumed that the cutting, bending, and welding techniques inside SEM will be widely used for fabrication, assembly of nanodevices and characterization of nanomaterials in the future.

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