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Direct Fabrication of the Scanning Probe Tip with Multi-Walled Carbon Nanotubes Using Dielectrophoresis

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ABSTRACT

We report a simple, low cost, and reliable method for assembling a multi-walled nanotube (MWNT) to the end of a metal coated scanning probe microscopy (SPM) tip. By dropping the MWNT solution and applying an electric field between an SPM tip and an electrode, MWNTs which were dispersed into a dielectric solution were directly assembled onto the apex of the SPM tip due to the attraction by the dielectrophoretic force. The effective measurement of a MWNT-attached SPM tip was demonstrated by direct comparison with AFM images of a standard sample with a bare AFM tip.

Key Words : Carbon nanotubes, Scanning probe, dielectrophoresis, AFM, Electric field

1. Introduction

Since introduced by Iijima[1] in 1991, Carbon Nanotubes (CNTs) have been used in many nanoscale applications due to their unique mechanical, electrical and chemical properties. Due to these specific characteristics, CNTs have been considered as a prime candidate in numerous micro and/or nanoscale sensors and devices such as field emitted displays, fuel cells, electronic circuits and mechanical sensors by Kong, Bachtold, and Martel[2-4]. Especially, distinctive mechanical properties of CNTs such as sharpness, high aspect ratio, high mechanical stiffness, high elasticity and metallic, semi-conducting characteristics have provided vast research and developmental opportunities in the application of CNT in the measurement of AFM images by the attachment of the CNT to the AFM tips by Stevens and Nagy[5,6]. Previous research for the CNT-attached AFM tip by Dai and Yenilmez[7,8] has been focused on CNTs fabricated by Chemical Vapor Deposition (CVD) method including the batch process for fabrication of CNT tips. It has been proved to be a very effective method since multiple probes with CNT at the apex of the tips can be produced simultaneously on a large sized wafer. However, there are some limitations such as non-uniformity of growing conditions and length of the CNT at the tip. A few attempts have been reported to attach CNTs on an AFM tip using the mechanical attachment method by Wong and Nishijima[9,10]. Lately CNT alignment on AFM tips was obtained using the magnetophoretic method or manual attachment after CNTs' deposition on electrodes using an electric field by Hall and Yamamoto[11-13].

In order to obtain a high resolution or chemically convenient modification, thin and short nanotube tip with

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single-walled nanotube might be required. However the multi-walled nanotube tip, which has about 10~100 aspect ratio in the case of CNTs with 10nm diameter, is useful to measure deep trench structures in semiconductor, bio and optic devices.

In this paper, we report a new attachment method of MWNT on SPM tips by utilizing the electrochemical method. This method is a simple, inexpensive and highly reproducible.

2. Experimental setup and physical explanations

An apparatus for aligning MWNT on the apex of an SPM tip is shown in Fig. 1. The apparatus consists of two parts; the processing part and the inducing part. The processing part consists of an Al electrode as a counter electrode and an SPM tip which is fixed to an x, y, and z axis translation stage for maintaining the distance with the counter electrode. The gap between the end of the tip and the surface of Al electrode is controlled by a translation stage within the micrometer accuracy.

The inducing part of the apparatus is composed of a signal generator and a power supply. The electric field can be generated by the ac component or the dc component. Through the ohmmeter, the gap between the end of the tip and the surface of the Al electrode can be closely controlled. It is very important to find the contact position between the SPM tip and the electrode, because we can measure the gap distance from that position. If the distance is too close, the end of a SPM tip is likely to be broken.

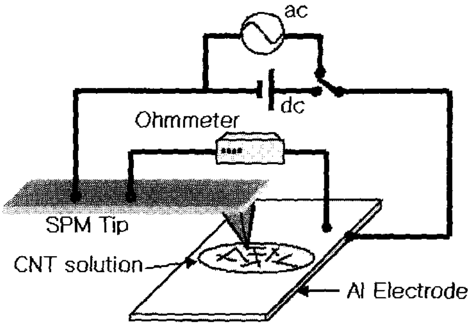


Fig. 1 Schematic of the electrochemical assembly apparatus with AC or DC voltage compatible electric circuit

On the contrary, if the gap is too far apart, the attraction force for nanotubes would not be high enough. In the experiment, the gap distance which has been determined based on the analysis of the electric field would be controlled within 10 ㎛.

The SPM tip was coated with Al 40 nm in thickness so that the apex of the tip became little blunter than a bare tip. The shape of the tip was still a cone type with full angle of less than 20°. As-produced MWNTs less than 10 μm length synthesized by an arc discharge method were used in this study. The outer diameter of the MWNTs is in the range between 10~20 nm. The MWNTs were suspended in ethyl alcohol and sonicated for about 1 hour so that the MWNTs could be untangled and homogeneously suspended in the solution. Before placing a drop of MWNT-suspended solution, the electric current was applied to the tip and the Al electrode. The typical volume of the MWNT solution applied for the assembly was about 10 ㎕ which contained enough MWNTs and sufficient amount of solution to prevent the solvent from drying out before CNTs are attached to the tip. The electric field was applied with DC or AC respectively. The frequency of AC field was in the range of 1~10 MHz, which was determined through systematic variations of the experimental conditions.

As discussed above, the attraction of MWNTs to the gap region and the alignment of MWNTs with an optimal orientation for assembly on the tip were accomplished by applying the electric field in an aqueous solution[12-13]. Several driving forces can be considered as a plausible explanation for the attraction and alignment of the MWNT. The MWNTs have a positive charge in ethanol, so they are attracted toward SPM tip(negative electrode) when we applied a AC voltage to the electrodes,. This type of driving force is created by the electrophoretic force. In the meantime, the applied DC voltage may also cause the dielectrophoretic force under the environment of non-uniform electric field.

In this letter, the most plausible explanation for MWNT tip assembling process is dielectrophoresis. This induced dipole, or polarization, can move, translate, and rotate a particle along the gradient of electric field. When an AC electric filed is applied to the electrodes, the polarizability has been induced on carbon nanotubes and a CNT goes through a dipole moment due to non-uniform electric field. The dielectrophoretic force which arises due to the non-uniform electric field can be expressed by the following equation[14-16].

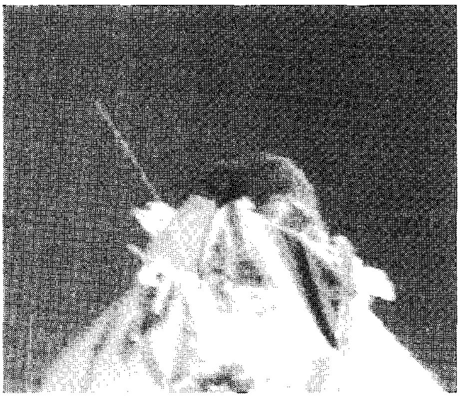
$$F_{DEP} = 2\pi a^3 \varepsilon_m \operatorname{Re}\left[\frac{\varepsilon_p^* - \varepsilon_m^*}{\varepsilon_p^* + 2\varepsilon_m^*}\right]\nabla|E|^2 \qquad (1)$$

where a is the longest dimension of the particle, ε_m is the dielectric constant of the medium, ε_p is the dielectric constant of the particle, and E is the electric field. This force is affected by the applied frequency due to the complex permittivity expression, $\varepsilon^* = \varepsilon - i\sigma/\omega$, in which σ is the conductivity and ω is the frequency of the AC electric field.

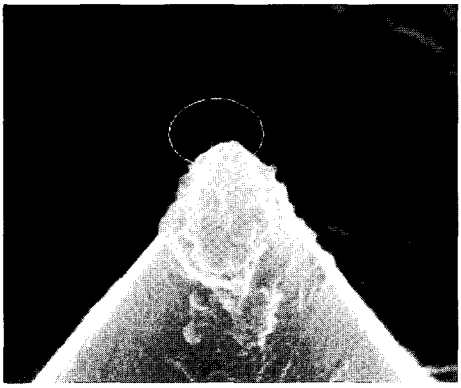
In the assembling process, particles with dipole moment moved toward the high density region of the electric field in a dielectric medium like ethanol. When we dropped the solution with nanotubes onto the gap between an AFM tip and an electrode plate, the particle with longer dimension and high dielectric constant was attracted first onto the highest electric field area, in this case, the end of the tip. Small and low dielectric particles slowly approach the highest field area. As a consequence, the positive dielectrophoresis is used for attracting the nanotubes. On the other hand, a particle with lower dielectric constant than the medium moves in the opposite direction, and this phenomenon is negative dielectrophoresis. According to this theory, in the case of the AC field, nano particles with the longest dimension have larger dipole moment, and therefore are first attracted and aligned to the tip than smaller particles such as carbon debris or other impurities. If the long and thin nanotubes attach onto the apex of the tip, smaller particles which arrive later at the tip cannot cover the nanotube completely. Therefore we can expect to obtain a protruding nanotube tip.

3. Experiments

We experimentally investigated the tendency of the MWNTs' attachment according to the AC or DC electric field only. When only a DC voltage was applied, impurities were overwhelmingly deposited on the tip more than the amount of deposited nanotube. Because impurities occupied almost the entire surface of the end of the tip, there might be no space for the deposition of nanotubes. If highly purified MWNTs are used in this process, a significant amount of CNTs could be deposited on the tip in spite of being under the condition of the DC field only. On the other hand, when only an AC electric field was applied, even though impurities were deposited on the tip, MWNT was attached on the end of the tip with a protruding shape. Several factors for assembling the nanotube were considered in this experiment. We found the major assembling factors such as the gap distance, applied voltage and frequency. Only when the applied AC voltage was larger than 5 V at the 10 ㎛ gap and 5 MHz, protruding nanotube could be found at the tip end. When the gap distance was over 10μm, the yield of the assembling was remarkably reduced. These results showed that the gap distance and the applied voltage were critical to attract the nanotube



(a)



(b)

Fig. 2 SEM image of CNT-attached SPM tips fabricated under an ac electric field with (a) 7 V_{rms} at 1 MHz, 10μm gap distance (b) 5 V_{rms} at 5 MHz, 5μm gap distance

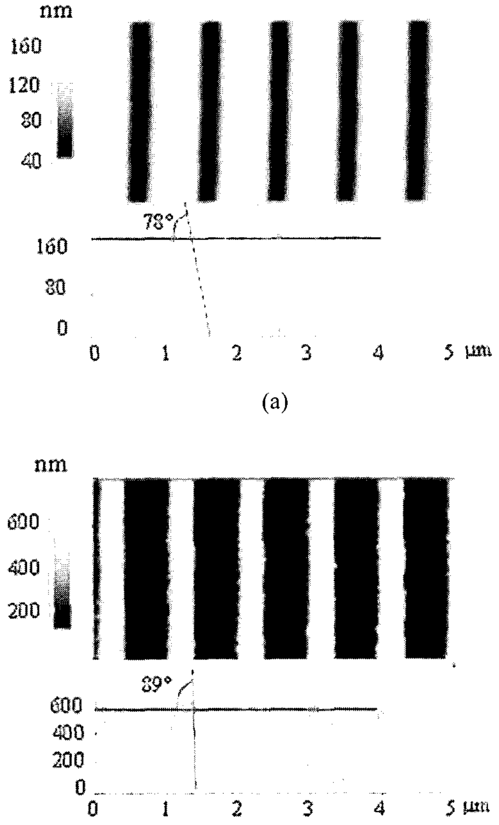
From the theoretical viewpoint, those factors must be related to the magnitude of dielectrophoretic force. When the frequency was varied from 100kHz ~10 MHz, there was little difference in the yield.

In these experiments, we observed several phenomena. At first, the orientation of nanotubes was always apt to be aligned parallel to the tip axis, because the attached part of the nanotube onto the end of the tip was guided according to the angle of the tip. We found that the angles of the obtained samples were in the range of 0~45° with respect to the tip axis. Next, when the current flowed through the nanotube (a tip would be often connected to an electrode due to nanotubes), the nanotube was burned and shortened. At that time, the current was about 10~20μA. As for the interaction between a nanotube and the tip, Van der Waals force may be the major force as shown in other studies[11-12], and additionally the impurities on the tip could induce firm attachment of the nanotube in our experiments. As for the protrusion of the nanotube, its maximum length would be always less than the gap distance, because the part of the nanotube which is longer than the gap would be used to attach on the tip area.

Using this method, it was possible to reproducibly fabricate MWNT-attached SPM tips with about 30 % yield. As shown in Fig. 2, MWNT deposited at the end of the SPM tip was observed by SEM and the contact area between the MWNT and the tip was covered by some impurities.

4. Performance evaluation

In order to demonstrate the effectiveness of the CNT-attached SPM tip in topographic measurement, we obtained the Atomic Force Microscopy (model: LS, PSI, USA) images of a standard sample by utilizing two SPM tips; one bare and the other a CNT-attached SPM tip. The standard sample used in this measurement was a patterned quartz plate with rectangular lines of 500 nm height, 300 nm widths and 700 nm of interline spacing.



(a)

(b)

Fig. 3 Tapping mode AFM images and line profiles obtained with (a) bare tip (158 nm height and 608 nm width) and (b) CNT-attached tip (500 nm height and 320 nm width)

The measured AFM images of the sample are shown in Fig. 3. The AFM images were obtained by tapping mode with a scan rate of 0.5 lines per second. As depicted in the AFM images, the cross-sectional line profile measured with a bare tip is significantly deviated from the real profile, whereas its profile measured with the CNT-attached tip resembles that of the standard sample. Furthermore, the measured dimensions of the height and width of the rectangular line with two different tips were significantly different. This result indicates that the lateral resolution was markedly improved by utilizing the CNT-attached tip.

5. Conclusions

We developed a simple, effective, inexpensive, and reproducible method for fabrication of the MWNT-attached SPM tip. It requires several minutes and no expensive apparatus. Therefore, this method can be used for research purposes as well as commercial use. The dielectrophoretic force generated by an AC electric field was used to attract and align MWNTs at the region of the tip and the counter electrode, and to allow their deposition at the end of the tip. Under this condition, we obtained CNT-attached SPM tips with about a 30% yield. By utilizing the CNT-attached tip, we could measure the topographic images of a highly trenched surface with higher accuracy and higher lateral resolution than a conventional tip. This method will provide the basis for mass production of the CNT-attached SPM tips with high yield and low cost. The next work will concentrate on controlling the orientation and length of the attached CNT and improve the yield of fabrication with a highly purified MWNT sample.

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