Direct attachment of carbon nanotube on scanning probe tip using dielectrophoresis

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ABSTRACT

We found the simple, effective and low-cost fabrication method of scanning probe tip with carbon nanotube. The assembling apparatus has been discussed and a plausible explanation about attachment mechanism based on dielectrophoretic force has been suggested. In order to find the proper assembling condition, electric field analysis for the round shape tip has been accomplished. Using this condition, the scanning probe tips with carbon nanotube were fabricated at 25% success rate.

Keywords: scanning probe microscope, carbon nanotube, dielectrophoresis

1. INTRODUCTION

To improve the imaging quality in vacuum, liquid, and special chemical environment, the role of Atomic Force Microscope (AFM) becomes important. The quality of information obtained from AFM tip depends on the size, shape and terminal functionality of the probe tips(1-2). Conventional commercial AFM tips have Si microfabricated cantilever and pyramidal shape with several ten degree of cone angle(3). So it is difficult to measure deep and narrow structure. An alternative approaches to resolve this problem took the sharpness of conventional tips using micromachining or deposition of amorphous carbon(4-5). The unique mechanical, electrical, and chemical properties of the carbon nanotubes (CNTs) make these ideal for scanning probe microscope. Carbon nanotube tips have several advantages, including high aspect ratio, low tip sample adhesion, high stiffness and resilience over 90 degree, chemically inertness, and stability in harsh environment like high temperature. AFM tip with single-walled carbon nanotubes (SWNTs) possible to measure the geometry with sub nanometer resolution due to their small diameter on the order of 1nm, while tips with multi-walled carbon nanotubes (MWNTs) are very suitable for measuring the deep trench structure because of their high aspect ratio with several um length. AFM CNT tips also have been considered as a tool for sensing the chemical signal, making the pattern lithographically, and for detecting the bio-molecule like as DNA(6). There are several method to make the CNT tips and now are trying to find more convenient and high productive method.

AFM CNT tips have previously been made by attaching nanotubes to the side of Si pyramidal tips under optical and electron microscopes(7-8). This method would be very good to control the assembling condition like as length or angle on the Si tip, but it would take long time to make. As one of recent study, Si tip picked up vertically aligned SWNTs grown from planar surface substrate. This method is based on exact observation using AFM. As direct as-grown method onto Si tip, chemical vapor deposition (CVD) methods also have been suggested(9-10). One of them use the growth of the catalyst onto the special location of the target tips, the nanotube grows using carbon gas in furnace. CVD enables the wafer scale production of the nanotube tips using semiconductor process, but still has several tasks to resolve. More recently, nanotube tips are being made at 50% success rate by attracting nanotubes into the end of the tip in solution using magnetophoresis(11). Each methods have unique advantages, and any of them has not seen as absolutely superior than the rest of them.

Here we report a new method to make the nanotube tips using dielectrophoresis, a major step is similar to the magnetophoretic method but more controllable and more simple. This method has two key steps for making the tips; one is to closely approach the distance between a AFM tip and a counter electrode, another is to attach the nanotubes onto the end of the tip using a solution which contains the carbon nanotubes and is dried out for a minute. Dielectrophoresis which attracts the particles in a medium with non-uniform electric field has previously been studied.
for aligning or separating of carbon nanotubes on the gap between surface electrodes (12-14). We directly uses the AFM tip as one of the electrodes. Many kinds of forces are likely to be applied to this process during attaching times, but the dielectrophoretic force is the most dominant factor which attract the nanotubes. We verified this result through the repeatable experiments.

2. EXPERIMENTAL SETUP AND THEORETICAL BACKGROUND

The experimental setup for the fabrication of the AFM CNT tips are shown as Fig. 1. Highly fine motion stage has been used for adjusting the distance between the tip and the electrode. A conventional AFM tips were coated with metal of good conductivity and used as a electrode itself. A counter electrode is also highly conductive metal, and it has flat surface and cleanness. Carbon nanotubes are sufficiently diluted with ethanol and sonicated for an hour in order to uniformly disperse in solution. Nanotubes thereby prepared were multi-walled, about 10nm in diameter and 5um in length. We used as-grown carbon nanotube which was made by arch discharge and includes much impurities. The reasons why we use the unpurified carbon nanotube is to demonstrate that anyone can easily make the carbon nanotube tips in one’s laboratory, because the MWNTs made by arc discharge is very difficult to purify. Alternating voltage of 7 Volt at 1MHz was then applied to the electrodes. With this experimental setup, the CNT was parallel aligned to electric field line, moved to the high electric field and attached the end of the tip with protruding shape.

![Concept diagram of CNT attaching mechanism with the scanning tip](image1)

**Fig. 1.** Concept diagram of CNT attaching mechanism with the scanning tip

![MWNTs without any purification process, grown by arc discharge](image2)

**Fig. 2.** MWNTs without any purification process, grown by arc discharge
The solution is volatile so that it dried out in a minute to avoid attaching too many nanotubes and impurities. A picture of a carbon nanotube sample has been taken by scanning electron microscope (SEM) as shown in Fig. 2. Many impurities are observed and few straight nanotubes can be found in some area as the partly tangled shape with impurities.

The dielectrophoretic force (DEF) is occurs from a dipole moment of the polarized particles in non-uniform electric field and can be written as the following equation(14).

\[ F_{\text{DEP}} = 2\pi a^3 \varepsilon_m \text{Re} \left[ \frac{\varepsilon_p^* - \varepsilon_m^*}{\varepsilon_p^* + 2\varepsilon_m^*} \right] \nabla |E|^2 \]  

where \( a \) is the longest dimension of the particle, \( \varepsilon_m \) the dielectric constant of the medium, \( \varepsilon_p \) the dielectric constant of the particles, and \( E \) electric field. The frequency dependent, complex dielectric constants shown with the asterisk are expressed by the combination of normal dielectric constants and conductivities (\( \sigma \)) shown in the following equation.

\[ \varepsilon_p^* = \varepsilon_p - \sigma_p \omega \] 
\[ \varepsilon_m^* = \varepsilon_m - \sigma_m \omega \]  

where \( \omega \) is the frequency of the applied ac electric field.

The most plausible explanation for AFM CNT tip assembling process is dielectrophoresis. This induced dipole, or polarization, can move, translate, and rotate an particle along the gradient of electric field. When ac electric filed is applied to the electrodes, carbon nanotube has induced polarizability and goes through a dipole moment due to non-uniform electric field. In this process, particles with dipole moment moved toward the high density of electric field in dielectric medium like ethanol. When we drops the solution with nanotubes into the gap between an AFM tip and an electrode plate, the particle with longer dimension and high dielectric constant firstly attracted into the highest electric field area. Small and low dielectric particles slowly approach to the highest field. As a consequence, the positive dielectrophoresis is used for attracting the nanotubes. We use the as-grown nanotubes with some of impurities which have larger dimensions than nanotubes so that the large impurities are firstly attached on the end of the tip. Of course, we know that it is difficult to expect high success rate, but there is a possibility that nanotubes with high aspect-ratio are finally attached onto the apex of the tip with protruding shape. If the long and thin nanotubes attach onto the apex of the tip, smaller particles which arrive at the tip later cannot completely cover the nanotube. So we can find the protruding nanotube tip.

### 3. SIMULATION FOR THE ELECTRIC FIELD

In previous section, important factors to deposit the nanotube were frequency, the strength of the electric field. Except for the above crucial parameters, there are several other parameters to affect the attachment of the nanotubes, such as a tip shape, the concentration of a CNT suspended solution, the drying-out time of a solution for deposition, and the quantity of a solution. Here, we analyze the electric field around the tip. We briefly modeled the geometry as 2-dimentional using electromagnetic finite element method. FEMLAB was used for simulation. As generally known, the end of the tip will has largest electric field density over the area of the electrodes, and the nanotubes will gather into that area. But we need to verify this result and get the value and trend of the electric field under several conditions. Especially, we modeled the tip as not sharp but blunt apex, because we use the conventional tip coated with a metal. We tried to compare this blunt tip, 100nm in radius with the sharp tip, 0 in radius. Obtained maximum nominal electric field for the sharp tip is 10 V/um while the blunt tip has 5.33 V/um when the distance between the tip and the electrode is 10um@5volts. As anticipated, the apex of the tip has the largest nominal electric field. At the end of any shape of tips, the line of the electric field is vertical to the electrode plate. It means the nanotube will be oriented to become parallel position with this electric line at the end of the tip.
In case of the sharp tip, the severely non-uniformed electric field near the tip end could be an obstacle to settle the nanotubes on apex of the tip. On the contrary, a round shape of the tip might give more stable area in electric field that nanotube can temporarily stay where the gradient of the square of an electric field is 0 (i.e. $\nabla |E|^2 = 0$) during the attachment. This region, however, is not an absolutely stable location. It is thus desirable to keep this region broad to keep the nanotubes stay longer. At the tip of the gap, the strength of the electric field was rapidly changed. The width of quasi-stable region on the edge of the gap was under 1 µm. From the fabrication point of view, the sharp tip is difficult to make, because the metal was coated onto the tip. On the contrary, round shape tip has broad stable region over 1 µm and also can be easily made by deposition a metal onto conventional Si tip. So we mainly simulated the round tip over several factors.

Fig. 2. Analytic results of electric field for (a) sharp shape tip with no radius (b) round shape tip with 10 um radius
The another important reason to vary the several analysis conditions is to find the relation between the factors and the strength of the electric field. As the major factors, we select the distance between electrodes and the magnitude of the applied voltage. In this simulation, a round tip only is considered. We simulated the electric field according to the variation of the gap distance between the tip and the electrode plate and showed the results in Fig. 3(a). It shows that the nominal electric field is almost linearly proportional to the applied voltage. And applying the high voltage also need special instrument and may be affect the nanotube. It is considered that 10 volts is a upper limitation. The electric field
according to the magnitude of the applied voltage also presented in Fig. 3(b). Because the useful gap distance is less than 10um, it is not easy to set the apparatus. If the gap distance becomes 1 or 10 mm, the experimental setup is relatively more easy. But the electric field is remarkably reduced and the applied voltage should raise up to several hundred volt in order to make similar electric field strength. That is not realistic. Based on the experimental setup limitations and previous studies about mobility of nanotube, 10um in gap distance and 7 V in applying voltage were selected as an proper experimental condition.

4. EXPERIMENTS

Fig.4. experimental result in cases of (a) 3Volt@ 5MHz (b) 7 Volts@ 5MHz. For each case, the gap distance between a tip and electrode plate has maintained to 10 µm.
Fig. 4(a) shows a failed tip when 3 volt@10 MHz was applied. In this voltage, a few impurities could be found at the tip end and regarded this as a weak attraction of the particles. We tried 5 volt @ 10MHz case, but we could not obtain the nanotube tips. More impurities than 3 volt@10MHz can find around the tip end. In the case of Fig. 4(b), the applied voltage went up to 7 volt@10MHz and obtained the nanotube attached tip as shown in circle. The protruding nanotube had 500nm in length at the apex of the tip, and the tip was covered with more impurities than the case of Fig 4(a). And we obtained the repeatable results at 25% success rate. Among reasons of the low yield, the most crucial factor might be the purity of the nanotubes. If we would improve the purity of nanotubes, we could expect better yield. Except for the purity, gap distance also should be constantly maintained.

We found the very simple, effective and reproducible method to make the AFM CNT tip, also explained the experimental setup and attaching mechanism, and suggested the proper assembling condition based on the simulated results for the electric field. Based on dielectrophoresis, The gap distance between a gap and electrode and the strength of the electric field were considered as the major factors which affect the attaching of the nanotubes. Considering the experimental limitation and previous studies, proper assembling condition was suggested and the nanotube tips were fabricated using the devised experimental apparatus. The CNT tip is very useful in nanotechnology as soft lithography tool, data storage unit and chemical or biology sensor. So this fabrication method will help to do the basic research using CNT tip in laboratory. And if we can improve the purity of the nanotubes, commercial CNT tip also can be fabricated at high success rate in the near future. This method can be extended to the assembly of the nanotube on the other substrate.

The authors thank to Arvind Raman in Purdue University and acknowledge help by Center of Nano Mechatronics & Manufacturing (Project #: 02-K14-01-008-2-0 ) as financial support of this work.

REFERENCE
