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RESEARCH NOTE

High Resolution Image with Multi-wall Carbon Nanotube Atomic Force Microscopy Tip

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ABSTRACT

In this paper, a simple and reproducible approach for attaching the multi-wall carbon nanotubes (MWNTs) to the apex of the atomic force microscope probe has been proposed. For this purpose, the dielectrophoresis method was applied due to its simple performance, cheapness and reliability. Here, specifically the effect of voltage on the deposition of MWNTs onto the tip of the atomic force microscope has been investigated while the other parameters held constant. Our experiments revealed that when the frequency was held at 5 MHz and 1 μL of MWNTs solution injected, the optimum voltage between tip and electrode surface was 6 V.

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1. INTRODUCTION

Since the first introduction of carbon nanotubes in 1991 by Iijima [1], they have been the major focus of intense research in the field of nanotechnology. These nanotubes consist of one atom thick carbon atoms rolled up into cylinders. There has been large volume of studies to exploit their unique mechanical, electrical and chemical properties, high aspect ratio, high strength and elasticity [2-4]. Amongst the wide applications of the carbon nanotubes is their use as tips in atomic force microscopy.

Atomic force microscopy has emerged as a powerful method for studying the surface of conductors, semiconductors, and insulators at nanometer scale. The ability to image and characterize structures in liquid, ambiant atmosphere and vacuum further increase its versatility. Because of its wide use, especially in harsh chemical environments, improving image clarification is of great importance [5-7]. In order to increase the quality of images, size and shape of probe tip can be changed. In conventional AFM tip, a Si or Si_3N_4 microfabricated pyramidal cantilever with high degree of cone angle and short aspect ratio with diameter about several tens of nanometers is used for probe structures. Theoretical studies have shown that the spatial resolution of AFM images is determined by two factors, the radius (sharpness) of the tip and its aspect ratio [8]. Recently, much effort has been made to improve the sharpness of AFM probing tips and to increase lateral resolution of AFM [9, 10]. However, problems still remain in obtaining high-resolution images since the Si or Si_3N_4 tips quickly become worn during operation and then tip profile changes occur, particularly when crashing the surface. As a result, imaging deep and narrow structures encounter difficulty. To solve this problem, we have another approach in hand, and that is carbon nanotubes. The unique mechanical, chemical, and electrical properties of the CNTs make them ideal for using as AFM tips. CNTs have some advantages such as low curvature radius and high mechanical strength [11-13]. Some other preferences of CNTs tip with respect to Si tip include high aspect ratio, high stiffness, low tip sample adhesion, and high resilience; also, CNTs are stable in harsh chemical environments and high temperatures. Their high aspect ratio enables the recording of high-resolution images of deep trenches in semiconductors, biological molecules, and stability in harsh environments like elevated temperatures. Depending on the diameter of CNTs, the clarity of images could be improved. Multi-walled CNTs have

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very low radius of curvature and help to increase the quality of images as high as several nanometers; On the other hand, multi-walled CNTs are suitable for measuring deep and narrow structures due to their high aspect ratio. In our previous studies we have succeeded to make single-walled CNTs tips. Now, in this work, we are going to construct multi-walled CNTs tips and investigate some effective important parameters of these structures.

Several methods have been investigated to either attach or directly grow CNTs on the apex of AFM probes [12, 14-19]. First one is the initial fabrication of a CNT probe by attaching a CNT bundle onto a cone-shaped tip, coated with an adhesive. Another method for CNT-attached AFM tips has focused on CNTs fabrication by chemical vapor deposition (CVD) method [20, 21]. However, those methods are costly and produce non-uniform results. Su reported the fabrication of a CNT probe using dc electrophoresis and electrostatic techniques [22-24]. We have used dielctrophorsis (DEP) as a convenient method for CNTattachment. This method is simple, inexpensive, and reproducible approach to obtaining a single CNT-attached AFM tips. Owing to reproducibility, reliability and cheapness, the periodic electric field (dielectrophoresis) technique for deposition of one carbon nanotube on Si tip is very suitable. In this method CNTs are polarized by a nonuniform electric field and then they are moved toward the highest electric area (end of the tip) by a positive DEP force. In this method, several factors for assembling the nannotube should be considered including applied voltage, gap distance, angle distance, concentration of multi-walled CNTs solution, and amount of a multi-walled CNTs solution. The assembling factors are highly interelated. The optimal assembling factors for making an AFM tip with a single multi-walled CNTs is obtained by repeated experiments.

Lee et al. reported that the attached nanotube should be short and parallel with the tip axis and distance between electrode plate and the tip apex of about 10 micrometers by this method [24]. We encounter two problems by setting the gap distance about 10μm. Firstly, it is very difficult to inject the CNT solution into the gap and secondly, it is possible that the upper part of the cantilever of the tip become immersed in solution. Since this part is used later to reflect the laser beam of the AFM, it should be clean. In this study, we increased the gap distance about 30μm and obtained the optimal assembling factors to fabricate a multi-walled CNTs-attached AFM tip.

2. EXPERIMENTAL

At first, the purchased multi-walled carbon nanotubes from "Chip-Tube" Company (which has been synthesized with CVD approach) should be purified. To do that, nanotubes were placed in a mixture of nitric acid (98%) and sulfuric acid (96%). This led to catalytic metals impurities present in the carbon nanotubes structure being removed. Then nanotubes were placed in a centrifuge machine. After gathering nanotubes at the bottom of container and filtering them, nanotubes were washed with deionized water. These processes should be repeated until the pH of the solution reaches at 7. These purified nanotubes were dispersed in deionized water with the aid of ultrasonic field for 30 min. Scanning electron microscopy image of multi-wall and purified carbon nanotubes are shown in Figures 1 and 2, respectively.

The schematic representation of experimental setup and dielectrophoretic force on MWNTs and charged particles in a nonuniform electric field are shown in Figures 3 and 4; including induced electric field and micrometer motion sections. The observation section includes an optical microscope (with magnification of 160X) and a CCD camera (to observe cantilever and tip). A smooth gold electrode, where a drop of nanotube suspension is placed on its surface and a signal generator has been used in induced electric field section.

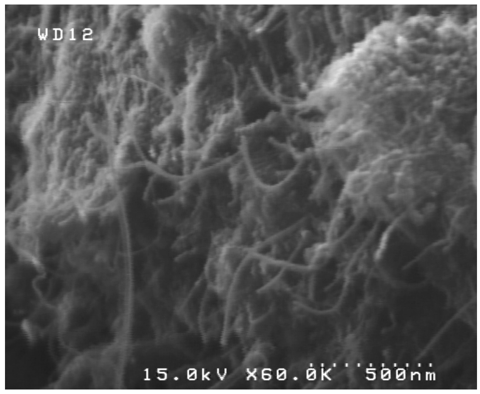


Figure 1. Scanning electron microscopy image of prestine carbon nanotubes

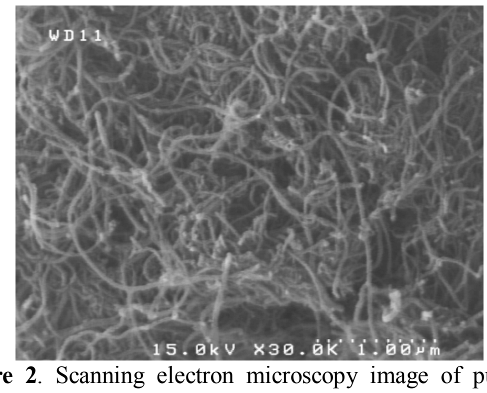


Figure 2. Scanning electron microscopy image of purified carbon nanotubes

In order to detect the current passed through and wave deformation, a multimeter and an oscilloscope were used respectively. Also, for displacement in microscale, a microposition machine with one micrometer accuracy was used. The gap angle is set by micrometer base in the rage of 0-90°. It was set at 20° in this experiment. The electric field can be generated by ac component. The distance between the end of the tip and the electrode plate can be controlled by oscilloscope. Nanotubes solution, which includes carbon nanotubes and deionized water, was injected in the space between gold electrode and tip by micropipette. Then, with the help of micro-position, tip comes down slowly until the space between electrode and tip arrives at 10 micrometer.

Several driving forces can be considered for the attraction and alignment of an MWNT. Dielectrophoresis, which is the translation motion of neutral matter caused by the polarization effect in a nonuniform electric field, should be carefully distinguished from electrophoresis which is the motion caused by the response to a free charge on a CNT in an electric field. Electrophoresis arises in both uniform and nonuniform electric fields. A charged particle is attracted to an electrode of opposite polarity in any electric field. In the case of a neutral particle, polarization occurs in a uniform and a nonuniform electric field but the particle will not move toward either electrode in the uniform electric field because the electric field intensity keeps its balance. A neural particle polarized in an electric field moves to the region of highest field intensity in a nonuniform electric field.

Figure 5 shows the optical microscope image of tip which has been entered into the solution. Note that we should be careful about the amount of nanotube solution drop, because we would have problem in reflected laser if back side of cantilever gets wet with the solution.

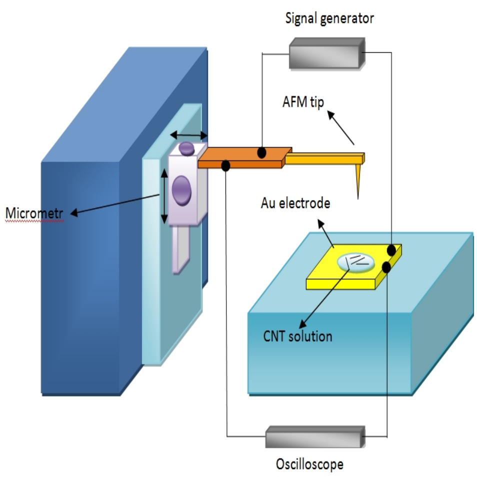


Figure 3. schematic drawing of experimental setup

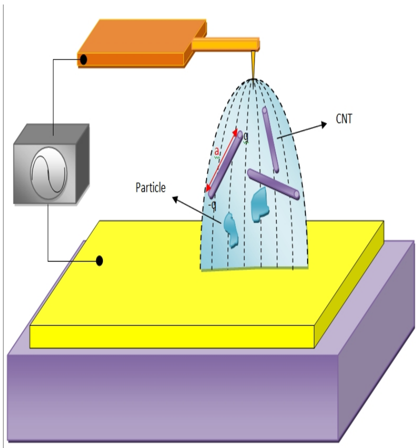


Figure 4. Dielectrophoretic force on MWNTs and charged particles in a nonuniform electric field.

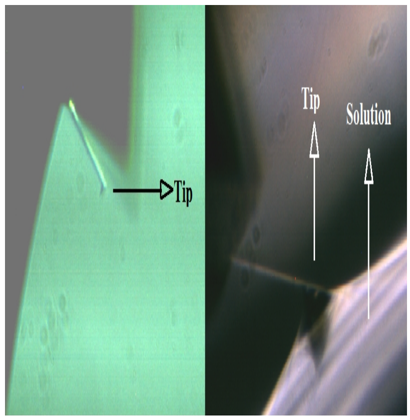


Figure 5. Optical microscopy image of the tip

Some parameters such as tip shape, distance between electrodes, deposition time and applied voltage should be concerned when the deposition of carbon nanotube on Si tip. Here, specifically the voltage effect on the deposition of MWNTs onto the atomic force microscope tip has been investigated while the other parameters held constant.

3. RESULTS AND DISCUSSIONS

From the physical viewpoint, carbon nanotubes resemble electric materials. Imposing an electric field on carbon nanotubes make them polarized. Because of high aspect ratio of carbon nanotubes, their polarization is more that the polarization of other carbon structures. If we apply a periodic electric field on carbon nanotubes, they start rotating and lead to the points with stronger electric field (from electromagnetics we know that electric field in sharp places is stronger compared to other places). Therefore, carbon nanotubes move toward the tip and attach its surface. There is a van der walls force between tip and carbon nanotubes (we have van der walls force between a conductor object and polarized dielectric object). Applied dielectrophoresis force on carbon nanotubes is as follows [25, 26]:

$$F_{DEP} = \frac{2}{3}\pi\, d^2\, l\, \varepsilon_m\, Re(K_A)\nabla|E|^2 \quad (1)$$

where

$$K_A = \frac{\varepsilon_p^* - \varepsilon_m^*}{\varepsilon_m^*} \quad (2)$$

$$\varepsilon^* = \varepsilon - i\frac{\sigma}{\omega} \quad (3)$$

In the above relations, l and d are the length diameter of carbon nanotubes, respectively, ε_m is the dielectric constant of the medium (here, water), and ε_p is dielectric constant of carbon nanotubes. $Re(K_A)$ is the real part of Clausius-Mossotti factor K_A, E is the electric field, σ is the conductivity, and ω is the frequency of the electric field. In addition to the imposed forces by electric field, multi-walled carbon nanotubes are subject to the thermal Brownian motion whose maximum value is acquired as:

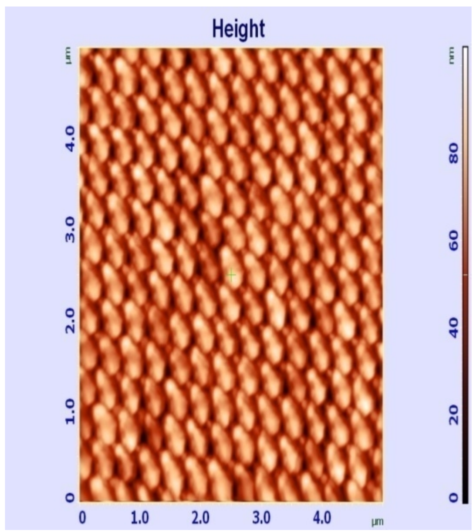
$$F_{Thermal} = \frac{k_B\, T}{d} \quad (4)$$

where k_B is Boltzmann constant, and T is the temperature. For $T = 300\,°K$ and 3 μm for length of carbon nanotubes, there is $F = 1.38 \times 10^{-15}$. The picture of scanning electron microscopy of conventional AFM cone-shaped Si tip is shown in Figure 6.

In order to induce conductivity to this tip and applying it as an electrode, a gold coating has been placed on it. Different voltages of 4, 6 and 8 V at 5MHz frequency was applied for 3s. Then, images of all samples were taken by scanning electron microscopy. From these images, it is realized that optimum voltage to make carbon nanotube tip for atomic force microscopy is 6 V. In voltage more than this optimum value, high amount of carbon nanotubes and other carbon structures including fullerene and amorphous carbon attach to the Si tip which is not desirable. In other hand, in voltage less that optimum voltage, the amount of attached carbon nanotubes to the Si tip during specified time was negligible. In Figure 6, image of as-made carbon nanotube tip in 6 voltages was seen. From Figures 6 and 7, change of curvature radius of carbon nanotube tip with respect to conventional Si tip is clearly observed. In order to investigate the quality of images taken with carbon nanotube tip, pictures from a standard sample taken by an atomic force microscope with carbon nanotube tip and conventional Si tip were compared. In Figures 8 and 9, the taken images from standard sample by AFM with both tips have been shown. From these images, it is realized that the taken images by carbon nanotube tip possess higher quality and give us information with more accuracy about the structure of sample.

Figure 6. Scanning electron microscopy of conventional Si tip

Figure 7. Scanning electron microscopy of CNT tip



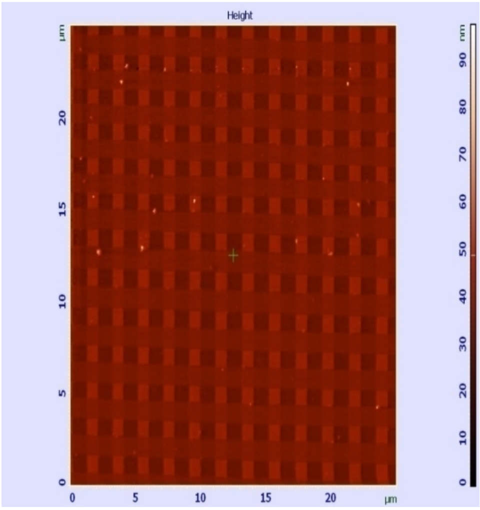


Figure 8. Atomic force microscopy image of standard samples with conventional Si tip

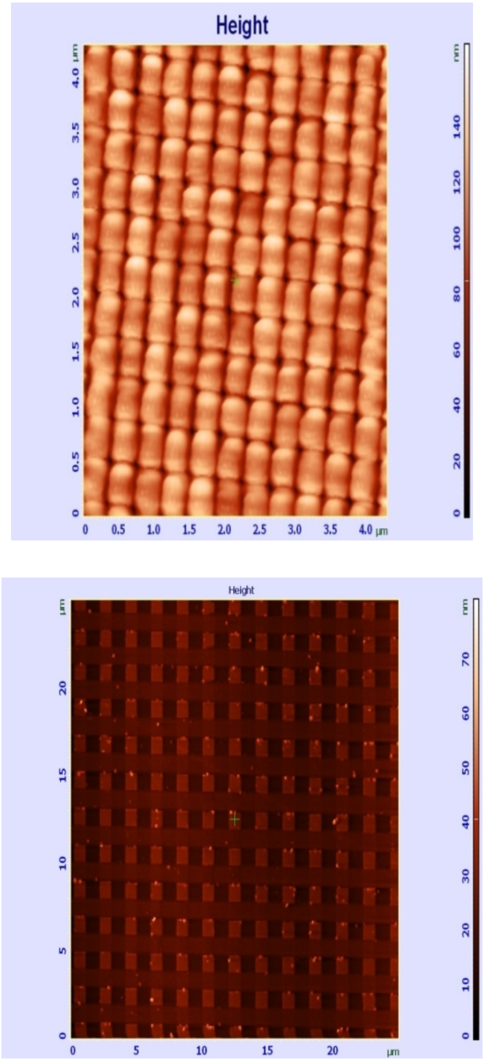


Figure 9. Atomic force microscopy image of standard samples with MWNTs tip

4. CONCLUSION

Using dielectrophoresis, we succeeded to attach a multi-walled carbon nanotube to the end of atomic force microscope tip. With the help of scanning electron microscope, the as-made tip has been characterized. Varying voltage and keeping other conditions constant, optimum voltage for this work was obtained as 6 volt in 5 MH_Z frequency, and a 10 μm gap between tip and gold electrode. Images taken by AFM with carbon nanotube tip show that these images have higher quality with respect to the conventional AFM and more accurate information can be resulted from these images.

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چکیده

دراین مقاله یک روش آسان و تکرارپذیر برای چسباندن نانولوله‌ی کربنی چند دیواره به انتهای سوزن میکروسکوپ نیروی اتمی ارائه می‌شود. روش به کار رفته در این تحقیق ، روش دی الکتروفورزیس است. این روش علاوه بر سادگی، بسیار ارزان و قابل اطمینان است. در این مقاله به طور خاص تاثیر ولتاژ را در شرایط ثابت بر نحوه‌ی انباشت نانولوله‌ی کربنی بر سر سوزن میکروسکوپ نیروی اتمی بررسی می‌شود. با توجه به آزمایش‌های انجام شده ولتاژ بهینه برای چسباندن نانولوله‌ی کربنی به سر سوزن 6 ولت/میکرومتر در فرکانس 5 مگاهرتز و تزریق ۱میکرولیتر محلول نانولوله‌ی کربنی در فاصله‌ی بین تیپ و صفحه‌ی الکترود می‌باشد.

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