2011 年第一届纳米操作、制造与测量（3M-NANO）

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长春理工大学电子信息学院（代章）

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Cutting Assembled Carbon Nanotubes with Atomic Force Microscope for Fabrication of Nanoelectrodes

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Abstract—This paper first presents a new fabrication method for CNT nanoelectrode pairs by combining the DEP and AFM lithography. The single CNT is driven and electrically connected with the microelectrodes by the DEP force, and then cut into nanoelectrode pairs through the AFM tip. The fabricated CNT nanoelectrode pairs can be used as probes to detect species in micro-environment and applied in electrochemical sensors.

Keywords—CNT, AFM, Nanoelectrodes

I. INTRODUCTION

Since carbon nanotubes (CNTs) were rediscovered by Iijima in 1991 [1], many scientists have done research on its synthesis, characterization, application in nanoelectronic devices and material field, such as transistors [2], integrated logic circuit [3], controlled growth [4], and sensors [5]. At present, there are three methods for synthesis of CNTs: arc-discharge, laser burning and chemical vapor deposition (CVD).

Although assembling the CNT to make devices is still a challenge, several groups have made some progress by different methods. Tian successfully assembled the single carbon nanotube (SWCNT) into the gap between two microelectrodes in 2008 [6]. Martin used patterned catalyst technology to directly grow CNTs in the device place [7]. Specially, multi-walled carbon nanotubes (MWCNTs) are a prospective material in nanodevices, which can be used as electrical connection in nanocircuit due to its metallic property.

Nanoelectrodes are ultramicro-electrodes, which can be used in ultrasensitive electrochemical sensors, or analytical tools for measuring electron transfer reactions and as probes to detect species in micro-environment [8]. Compared to microelectrodes, nanoelectrodes can break through the conductivity limitation and be directly used in measurement for electroanalysis, while microelectrodes need electrolytes to adjust the conductivity, pH and so on. Generally speaking, nanoelectrodes can provide higher mass transferring rate, less time constant, higher signal-to-noise ratio, better operability, higher sensitivity and greater capability. Due to its perfect properties, nanoelectrodes can be used in electroanalysis, sensors, nanoelectronics and so on.

So far, there are four methods to make nanoelectrodes, which are template making, etching method, self-assembly and lithography method. As CNTs have good conductivity, electron transfer ability, biocompatibility and good chemical stability, it is the best choice for making nanoelectrodes. Yi Tu and his group fabricated CNT electrodes with lower capacitive current by spin-coating of epoxy resin and used it in detecting ion concentration [9]. Xiang Wei and his group used MWCNT-graphite paste electrodes to detect Pb²⁺ [10]. Li Jun detected DNA by nanoelectrodes based on MWCNT with high sensitivity, which could be used for molecular diagnosis due to their well defined nano-scale geometry [11]. While in making nanodevice based on CNT nanoelectrodes, how to accurately control CNT's position and its size is a big problem. And limited by disperation technology of CNT bundles, most studies are on CNT bundles based nanoelectrode, which limit the performance of nanoelectrode and then the nanodevice. Thus, how to fabricate individual CNT-based nanoelectrode becomes very important and urgent. Shen and his group has successfully fabricated individual nanoelectrode based on MWCNT by SEM, while using a probe to pick up a CNT is not an easy job [12].

To solve the above proposed difficulties, in this paper, the authors will assemble the single MWCNT into gaps between electrodes, and then mechanically cut it into pairs by the AFM system. Also the CNT based nanoelectrodes will be characterized by the AFM system.

II. EXPERIMENTAL MATERIALS AND SYSTEM

A. Materials

MWCNTs, whose average diameter is 5-25nm, and length is 1-4μm, were synthesized by CVD. The 10μm wide electrodes with a 3μm wide gap were made by lithography.

B. Experimental system

The experimental system for assembling CNTs is our custom-built Di-electrophoresis (DEP) system, while imaging and manipulating of CNTs are implemented by Dimension 3100 AFM with a Nanoscope IV controller.

C. MWCNT suspension preparation

To prepare an individual MWCNT suspension, the mixed solution of MWCNTs and sodium dodecyl sulfate surfactants (SDS) (the concentration of MWCNTs and SDS is respectively 0.1% and 1%) was stored in a test tube and the tube was sealed by a plastic film. Then put the tube in the ultrasonic device and sonicate it for 2h at 59 KHz and 35 ℃. Next, the solution was centrifugated at 20000g for 0.5h to remove impurities and MWCNT bundles. The upper solution was the well-distributed MWCNT suspension.

D. Assemble MWCNT by DEP

During the MWCNT assembly, a 10V and 2MHz AC electric voltage was applied on the microelectrodes. The
detailed process is: (1) 1.5μl MWCNT suspension was transferred to the gap of microelectrodes by the liquid-transferring gun; (2) the voltage was applied and last for 15s; (3) the chip was rinsed in de-ionized water to remove SDS; (4) the chip was kept at room temperature for a while to evaporate the residual water.

E. Image and manipulate the CNT

When the chip was dry, an AFM based nanomanipulation system was used for imaging and manipulating of CNT. The work was done at room temperature in air. The probe used here for imaging is model MPP-11100 with a rectangle Si cantilever whose resonance frequency is about 300 KHz and force constant is 40N/m, and a tip whose radius is less than 10nm. And imaging is under tapping mode. The probe used for manipulating is the diamond-plated probe (MPP-11150-10 from Veeco) with a cantilever whose resonance frequency is about 140 KHz and force constant is 80N/m, and a tip whose radius is 25nm.

In the paper, the author used two methods to cut the single MWCNT. In the first method, the probe was contact with the substrate followed a path to make MWCNT ruptured (Fig. 1a). In the second method, the probe moved along the z axis, and gave the MWCNT a pressure to break it (Fig. 1b).

Fig. 1. Principles of cutting the MWCNT. (a) is the first method that the probe contacted with the CNT and followed a path and (b) is the second method that the probe moved along the z axis

III. RESULT AND DISCUSSION

A. Image of MWCNTs between microelectrodes

Fig.2 shows the different results for DEP assembling. MWCNTs were assembled between microelectrodes at 2MHz (Fig. 2g), 4MHz (Fig. 2c and Fig. 2e) and 8MHz (Fig. 2a). The data scale is 100nm. From the cross sections, the height of CNTs are about 5-20nm. There is no obvious difference in diameter and straightening of CNT at different frequencies from 2M to 8M. It proves the frequency is not an important influence on assembling of CNTs by DEP in a certain range. This result is in accordance with the DEP theory [13]. The number of MWCNTs in the gap was controlled by the concentration of CNTs and the signal duration.

B. Mechanically cut the MWCNT
When the MWCNT was cut into two parts, they can be used as nanoelectrode pairs, and the gap between them is in nanoscale.

The third MWCNT counted from the left in Fig. 2a was first cut along the path indicated by the black arrow (Fig. 3a). Due to the MWCNT has a large Young's modulus, it is very flexible. To cut it off, the probe moved along the path for several times with a velocity of 1 μm/s in x-y direction and 0.5 μm/s in Z direction. During this experiment, the AFM system was connected to an oscilloscope to see the cantilever oscillation signal. If there is a saltation, the CNT is cut off. The uncut and cut CNT are shown in Fig. 3a and Fig. 3b. Fig. 3c is the large-range scan image including the microelectrodes and the gap between them. Meanwhile, the gap between the cut CNT was measured to be 330 nm wide. In fact, the MWCNT is not really cut off but pulled off. So the probe velocity should not be too small and the path must be long enough to pull it off. If the path is very short, the MWCNT is just pulled and becomes deformed. There are experiments in low velocity and short probe path. Fig. 4 was conducted along a short path at a low velocity, the MWCNT is just deformed. Due to there is thermal drift in this system and the MWCNT has a very well flexibility, the short probe path lead to too small force to pull the MWNT off.

Although the above method is very effective for cutting nanowires, it is not the best. When cutting, the size of the gap is not easy to control and broken position is not definite since it is pulled off. It may be broken in defective parts. Based on the above issues, we have presented another method. In this method, we controlled the movement of the probe in Z

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Fig. 2. CNT assembly, the data scale is 100 nm; in (a) MWNTs were assembled between electrode under the frequency of 8 MHz; in (c), (e) the frequency is 4 MHz; in (g) the frequency is 2 MHz; (b), (d), (f), (h) are the cross section of the gap in the last graph.

As MWCNTs have good metallic property, they can be used as connection in electric devices. With an AFM manipulation system, the MWCNT will be cut mechanically.
direction. When cutting, choose a location in MWNT, and control the downward movement of the probe, repeating the motion for several times, the MWNT is cut off finally. In Fig.5, the gap in circle is cut by the first method and the gap in rectangle frame is cut by the second method. The second method is just like cut the MWNT use a knife. Because drift exists, several nuzzle points must be chosen as the probe location, so it is time consuming.

IV. CONCLUSIONS

In this paper, for making CNT nanoelectrode, DEP technology is used to assemble MWCNT into the gaps between microelectrodes after well-dispersed treatment. And the figs obtained by AFM proved that CNTs were successfully assembled. After that, AFM nanomanipulation system is used to mechanically cut MWCNT to get nano gap, which can be used as nanoelectrode. With the method and technology proposed in this paper, we can make single CNT based nanoelectrode which is important for making nanodevice, such as sensors for molecule detecting and etc. Meanwhile, for accurately controlling the gap size, there are still some work left to be done, for example, the path and velocity of AFM probe should be optimized and etc., and in the next step, the research will be focused on it for getting more accurate gap size.

REFERENCES