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Magnetic Force Microscopy using fabricated cobalt-coated carbon nanotubes probes

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Abstract

Magnetic force microscope (MFM) is a powerful technique for mapping the magnetic force gradient above the sample surface. Herein, single-wall carbon nanotubes (SWCNT) were used to fabricate MFM probe by dielectrophoresis method which is a reproducible and cost-effective technique. The effect of induced voltage on the deposition manner of carbon nanotubes (CNT) on the atomic force microscope (AFM) tip was investigated. The optimum voltage and frequency of SWCNT solution are obtained as 13 volts and 2 MHz, respectively. After coating theas-prepared CNT tips with a layer of cobalt,it can be used to obtain high resolution MFM images.

Keywords: Magnetic force microscope; dielectrophoresis; carbon nanotube probe; MFM probe.

Introduction

Scanning probe microscopy (SPM) has become a standard technique for obtaining two and three dimensional topographical images of surface with atomic resolution [1-4]. In addition, it may be used in many applications such as investigation of mechanical, chemical, electrical, magnetic and optical properties of surfaces, study of friction and adhesion forces, modifying a sample surface, crystal growth study and process controlling [4-12].

The ability to image and characterize structures in liquid, ambient, UHV, organic

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solvent vapour and biological buffer further increase their capability and making it possible to observe the system in states that are simply inaccessible to other techniques with comparable resolution and they can be also used over a wide range of temperatures[4, 13-14].

The interaction between the sample and the tip of a scanning probe microscope will vary depending on differences of the forces, which derived from van der waals, magnetic, double layer, electric, adhesion and capillary interactions. Therefore, SPM has been operated in different modes [15]. The invention of SPM techniques such as, scanning tunneling microscopy (STM, 1982), AFM (1986) and subsequently, MFM (1998) caused a revolution in surface science. By these techniques, the surface images are obtained by mechanically scanning the sample surface and recording the probe-surface interaction as a function of position. Thus, observation of surface structures in nanometer scale and real space is realizable [5,16].

MFM is a scanning technique, which derived from atomic force microscopy, where a magnetized tip scans a magnetic sample; Changes in magnetic force between tip and sample are sensed by the tip which is attached to a flexible cantilever and used to investigate the magnetic domain structure of sample surface in nanoscale level, and is widely used in the research and development of magnetic nanodevices [17].

Magnetized tip at one end of a flexible cantilever; generally an AFM probe with a magnetic thin coating ~< 50 nm magnetic film, such as Ni or Co, with typical 40 nm curvature radius of the tip is used as a commercial MFM probe. These probes with various coating thicknesses are used to achieve different magnetic moments which will be suitable for measuring of various samples. The size and shape of the probe tip can affect the quality of MFM images. There is the possibility of image artifacts, which could be induced by an unsuitable tip [18]. For imaging deep and narrow structures, there is difficulty when commercial tips are used and sharp tips are needed. Carbon nanotubes probe with low curvature radius and high mechanical strength can be used for this purpose [19].

The advantage of using carbon nanotubes as probes for SPM have been recognized for many years, it was known that the shape of CNT and their mechanical, chemical, and electrical properties make them ideal for using as SPM Probes.

Some other benefits of CNT tip with respect to commercial tips include high aspect ratio, stiffness, Young's modulus, elasticity, strength, and stability in harsh chemical environments and high temperature.

Various methods have been developed for fabrication of SPM carbon nanotubes based probes, which divided into two major groups: gluing or direct attachment method and direct growing the CNT on the SPM tips [20].

MFM probes can also be fabricated by these methods: mechanical attachment of metal-filled CNT and catalyst-attached CNT under SEM observation, and coating the CNT probe with a layer of magnetic metal [21-27]

Su et al. reported the fabrication of a CNT probe by using dc electrophoresis and electrostatic techniques [28-30]. Owing to reproducibility, reliability and cost effectiveness of the periodic electric field (dielectrophoresis), this technique is suitable for deposition of carbon nanotubes on AFM probes [19].

In this work, we attached CNT to the metal coated AFM probe by dielctrophoresis method and then magnetized it by coating with a Cobalt layer using sputtering technique. Then, some structurally effective and important parameters have been investigated. The fabricated probes have been characterized by SEM technique. MFM high resolution images obtained with fabricated CNT probes and compared with commercial probes.

Experimental

SWCNT with a mean diameter of 3-10 nm and a length of up to several micrometer have been prepared using a standard catalyst chemical vapor deposition (CVD) method over Co-Mo/MgO catalyst by methane decomposition at 600- 900°C in the Research Institute of Petroleum Industry [31]. The 'as-produced' materials contain amorphous carbon and catalyst particles (Co-Mo-MgO). In order to purify the tubes, acid treatment was performed. CNT were heated in an oven at 260°C for 30 minutes, followed by cooling to room temperature. Afterwards, they were mixed and stirred with 6M HCl for 16 h at 50 °C, followed by filtration and washing for several hours with distilled water for adjusting to the neutral pH of 7. Then the CNT were mixed and stirred with 3.5M HNO_3 at 60 °C for 3 h. Subsequent filtration and washing for several hours with distilled water led to purified nanotubes, which after drying in an oven at 100°C can be used to fabricate MFM probe using dielectrophoresis method. The morphology of SWCNT was studied using transmission electron microscopy (TEM). Figure 1 shows TEM image of purified SWCNT which was recorded by CM260-FEG-Philips microscope.

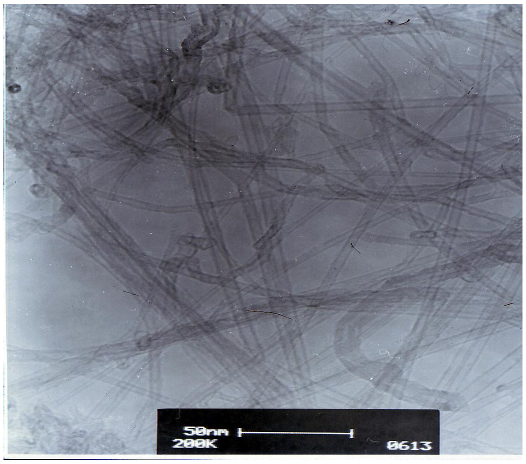

Figure 1.TEM image of purified SWCNT.

The experimental setup (Figure 2) is similar to what we have reported previously [19] which consists of different parts. A commercial Si AFM probe coated with an Au thin layer with 30 nm thicknesses and a small smooth gold ring were used as the working and the counter electrodes, respectively. The system consists of a micro-position machine which can alter the position of AFM tip in three dimensions with submicrometer resolution and adjust the distance between AFM tip and electrode plate. The gap angle is set by micro-position machine in the rage of 0° and 90°, which in these experiments was set in 20° as optimum angle. Signal generator can be used to generate AC electric field. An oscilloscope is used to detect the current passed through and control the distance between the end of the tip and the electrode plate.

The observation part consists of an optical microscope which was equipped with a charge-coupled device (CCD) camera connected to a computer for monitoring and recording the process.

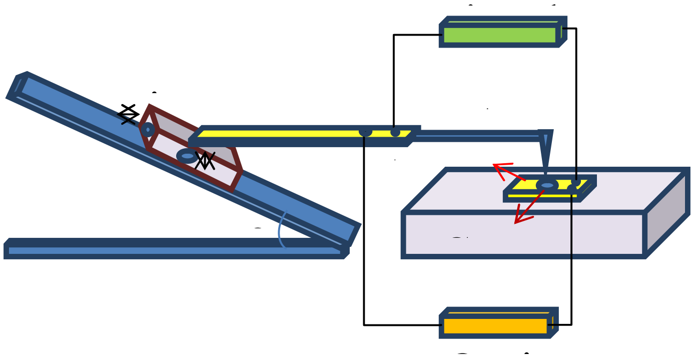

Figure 2. The experimental setup

SWCNT were diluted with deionized water and sonicated for 30 min in order to obtain a uniform dispersion. A droplet of SWCNT suspension (about 2 or 3 µl) was injected into the space between gold electrode and tip by micropipette. Then, with the aid of microposition machine, tip comes down slowly until the space between electrode and tip arrives at 30 µm.

It should be noted that the amount of SWCNT solution that is casted is very important. The contamination of the back side of the probe with the solution can change the path of laser beam and causes difficulty in imaging. After attaching CNT to the apex of metallic AFM tips, a layer of cobalt was sputtered on the CNT preattached tips.

Some parameters such as tip shape, the distance between electrodes, density and concentration of solution, deposition time and applied voltage should be concerned when depositing SWCNT on Si tip. Here, while the other parameters held constant, the voltage effect on the deposition of SWCNT onto the AFM tip has been investigated as an important parameter.

The tip used in this experiment was in tetrahedral shape (model NSG10). Tip curvature radius, cantilever length and the chip dimensions were 6 nm, 95 µm and 3.4×1.6×0.3 mm, respectively.

After immersing the tip into the SWCNT solution, a non-uniform field is applied to the solution for 4 seconds by a power supply which is adjusted in various voltages ranging from 11-15 volts and a frequency of 2 MHz, and then the probe is removed from the solution.

It should be noted that due to the rapid evaporation of the solution, testing should be carried out in a short time interval.

For the probe to be magnetic, it should be coated with a thin layer of cobalt with a thickness of about 30 nm using a sputtering device. The diameter of the resulted layer is governed by the current-time relationship.

A commercial scanning probe microscope (Solver P47H, NT-MDT Company) operated in MFM mode was used to make images from magnetic samples.

In order to achieve more toughness to the fabricated carbon nanotubes tip after attaching it, the annealing procedure can be carried out.

Results and discussion

AC electric field applied to the electrodes polarizes the polarizable particle which is suspended in a non-uniform electric field and a force is exerted on dielectric particles which is called dielectrophoresis. The dielectrophoretic force that arises due to nonuniform electric field is attractive or repulsive depending on the orientation of the dipole.

Appling an alternating electric field on the carbon nanotubes causes rotating them and leads to the movement toward the sharper points with stronger electric field (the electric field in sharp places is stronger with respect to the other places). Therefore, carbon nanotubes move toward the apex of tip and attach to its surface. There is a van der Walls force between tip and carbon nanotubes (a van der waals force can be seen between a conductor object and polarized dielectric object). Applied dielectrophoretic force on carbon nanotubes is as follows [32, 33] .

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

Where *l* is the length of carbon nanotube, d is the diameter of carbon nanotube, ε_m is the dielectric constant of the medium (here, it is water), ε_p is the dielectric constant of carbon nanotube, Re(K_A) is the real part of Clausius-Mossotti K_A factor and *E* is the electric field. This force is related to the frequency of the applied electrical field because of the complex permittivity is frequency-dependent based on the following equation:

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

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

Where σ, ω and i are electrical conductivity, the frequency of the electric field and imaginary unit, respectively.

In Figure3, scanning electron microscopy image of conventional AFM Si tip is shown in tetrahedral shape.

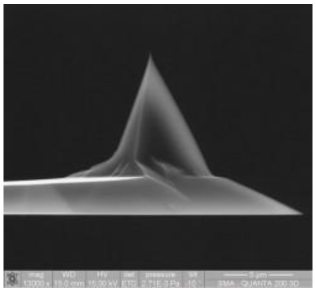

Figure 3. Scanning electron microscopy of conventional Si tip

Some parameters such as characteristics and concentration of CNT, time, applied voltage, frequency, gap angle and distance affect the attaching CNT to Si gold coated tip.

Carbon nanotubes with the longest dimension have larger dipole moment. Therefore, these attached to the tip faster than smaller particles.

If the concentration of CNT or the time during which attaching of CNT performs were too high, the bundle of CNT attached instead of a SWCNT to the AFM tip apex.

The gap distance is an important parameter in manufacturing CNT tip; low gap distance causes difficulty in handling the experiment and introducing the CNT suspension droplet into the gap of the electrodes.

In this work, the gap distance is increased up to 30 micrometer and the effect of voltage on attaching CNT is investigated in constant frequency, time, concentration, gap angle and distance.

Various voltages of 11, 12, 13 and 14 V is applied in constant frequency, time, concentration, injection volume of a SWCNT solution, gap angle and distance of 2 MHz, 3 s, 0.01 g/L, 5 µL, 20 Degree, and 30 micrometer, respectively.

Pre-attached CNT tips images were taken by a Scanning electron microscope XL-

30 Philips with EDX analysis.

Results showed that the optimum voltage for making CNT tip is 13 V, while other parameters held constant in 30 micrometer gap distance.

Higher voltages cause high amount of CNT be attached to the tip and in voltages less than 13 V, the amount of attached CNT was negligible.

Figure 4 indicates the SEM image of as-made CNT tip in 13 V in which an attached CNT on the apex of the tip is clearly realized. Then, suitable pre-attached CNT tip was coated by Cobalt with a thickness of 30 nm for inducing magnetic property to the tip.

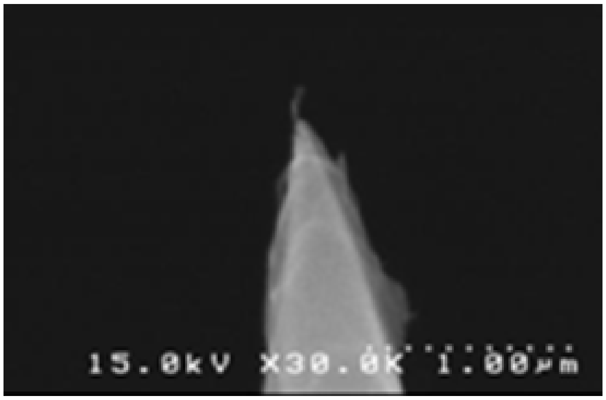

Figure 4.SEM image of CNT tip

In order to investigate the quality of CNT tip, magnetic force microscopy images were prepared using SPM in MFM mode.

Magnetic samples images were made using MFM standard and magnetic CNT probes and results were compared.

MFM images from hard disk with both tips have been shown in Figures 5 and 6 which realize that the taken images by CNT tip with higher resolution have more quality and give us information with more accuracy about the structure of sample and magnetic domains.

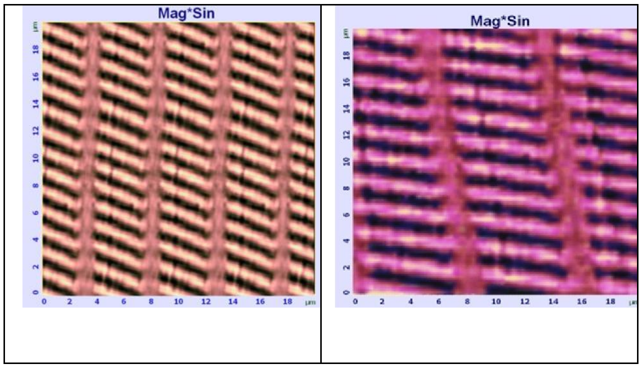

Figure 5. MFM image prepared with CNT probe

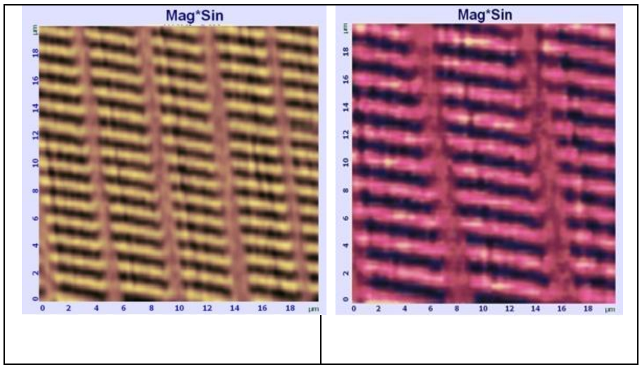

Figure 6. MFM image prepared with MFM standard probe

Conclusion

SWCNT were used to make MFM probe using dielectrophoresis technique. Concentration of CNT, time, applied voltage, frequency, gap angle and distance are parameters which affect the attaching of CNT to the Si gold coated tip.

Gap distance is also an important parameter in manufacturing CNT tip; handling the experiments in higher gap distance between electrodes can be easily performed.

In this work, the gap distance is adjusted in 30 micrometer and the effect of voltage on attaching CNT is investigated while the other parameters such as: frequency, time, concentration, gap angle and distance held constant.

The results of this work indicated that the optimum voltage for making CNT tip is 13 V, when the frequency, time, concentration, injection volume of SWCNT solution, gap angle and distance having constant values of 2 MHz, 3 s, 0.01 g/L, 5 µL, 20 Degree, and 30 micrometer, respectively.

High resolution images obtained with the proposed cobalt coated CNT tips with respect to the conventional MFM probes are indicative of good performance of the constructed tips and it can be said that more accurate information can be resulted from these images.

Acknowledgments

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