Fabrication of individual aligned carbon nanotube for scanning probe microscope

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Abstract. This paper demonstrates the fabrication of a micro-cantilever equipped with a high aspect ratio carbon nanotube (CNT) tip to perform surface characterization with high spatial resolution. A single vertical CNT, synthesized on a freestanding tip-less micro cantilever, is intended to be used for scanning probe microscopy (SPM). E-beam lithography and lift-off technique was adopted to define a nano-sized catalyst particle. An individual CNT was then synthesized by using an inductively coupled plasma chemical vapor deposition (ICP-CVD) system. The freestanding individual aligned CNT probe was then used to characterize a silicon nano pillar structure. The surface morphology was scanned in a contact mode and 0.1 nN set-point force. The ultra fine resolution can be characterized without wearing the probe and sample. The CNT probe shows a great potential as a high sensitive sensor operating in an optimally adjusted atomic force microscopy (AFM) system.

1. Introduction
Scanning probe microscopy (SPM) has become a powerful tool in nano science and technology due to its unique abilities in imaging, characterizing and manipulating nanometer-scale structures [1-2]. The surface morphology can be imaged in atomic level, revealing not only the structure characteristics, but also the distribution of defects and impurities. Therefore, the development of probe fabrication processes and tip sharpening methods has caught an intensive attraction recently [3-4].

Recently, high aspect ratio carbon nanotubes (CNTs) have demonstrated a great potential becoming an ideal material for the SPM tip because of its well defined geometry, large Young’s modulus, and robust mechanical/electrical properties [5]. The CNT probe can reversibly buckle under high load without wearing, so that the damage to both tip and sample can be greatly reduced. The methods of attaching a CNT on the probe tip can be classified into two categories: one is the mechanical mounting technique and the other
is the self-assembly method by direct chemical vapor deposition (CVD) synthesis [6-7]. In the former approach, a CNT must be assembled one by one onto the probe tip, which is time-consuming, tricky, and not suitable for mass production. The direct growth approach is in principle with no mass production limitation. The technique is, nevertheless, not yet readily available to synthesize an individual and well aligned CNT on the tip apex of the probe, with good controllability in the diameter and length.

2. Methods and Experiments
To synthesize a single CNT on a SPM probe as a tip, the size of the deposited catalyst on the apex of the silicon tip is a critical issue. To avoid the difficulty in growing a single CNT on silicon tip, the idea is to grow CNT directly on the silicon nitride cantilever beam to replace conventional silicon tip. This paper proposes a method of applying E-beam lithography (EBL) to define the catalyst pattern on a designated position of tip-less cantilever beam structure. E-beam lithography has been a primary technique for defining, patterning and connecting experimental structures is nanoscale. Silicon nitride cantilever arrays are fabricated by silicon bulk micromachining technique. An individual aligned CNT is grown by an inductively coupled plasma CVD system onto the nano-sized catalyst spot defined by EBL near the free-edge of the micro cantilever structure. A field emission scanning electron microscopy (FE-SEM) was used for examination.

2.1. Fabrication of silicon nitride cantilever arrays
The freestanding cantilever arrays were fabricated by micromachining processes, as illustrated in figure 1. The substrate was double-side polished p-type (100) silicon wafer of 400 µm thickness. A 1.5 µm thick layer of low stress silicon nitride (Si₃N₄) was deposited on each side of the substrate by Low Pressure Chemical Vapor Deposition (LPCVD). A 250 µm long and 50 µm wide silicon nitride beam was patterned by photo lithography and reactive ion etching (RIE) for using as the cantilever structure. Next, nano-scale dot hole was made by E-beam lithography and catalyst metal was deposited by lift-off technique. The position of the nickel dot was defined by E-beam writer on poly-methyl-methacrylate (PMMA) with a thickness of about 35 nm. The exposure dwell time of E-beam was 8 ms under 6 nA current with 20 KV acceleration voltage. After development, the 10 nm nickel thin film was deposited by E-beam evaporation, and the catalyst patterns are then defined by the followed lift-off process. To protect the front side of the substrate, the substrate were then placed in the CVD chamber for Parylene C coating. On backside through-wafer etching, 35% KOH solution was employed at 75°C to release the cantilever. Isopropyl alcohol (IPA) was added into the KOH solution to reduce etching undercut. After through-wafer back-side etching, the residual Parylene C was removed by O₂ plasma and the cantilever array was then revealed. Figure 2 shows the FE-SEM images of a freestanding silicon nitride cantilever array.

Figure 1. Fabrication flow chart of an individual freestanding CNT tip on cantilever probe.
2.2. Carbon nanotube growth with ICP-CVD process

The CNTs synthesized by arc-discharge or laser ablation are usually randomly tangled mixed with impurities [8-10]. Normally, CVD is a batch process to grow pure and dense CNT arrays on designated position defined by catalyst metals. In this research, the individual vertically aligned CNT was grown by using an ICP-CVD system, and the growth condition was optimized on the silicon substrate. Briefly, an ICP-CVD system was used to grow CNTs under the following process conditions: ICP power of 1000 W, substrate RF bias of 300 W, feed gas mixture of C₂H₂/H₂/Ar with 8/24/0.5 sccm flow rates, and total pressure of 20 mtorr. The substrate temperature was about 550°C and the growth time was 10 minutes. Figure 3 shows array of well-aligned free-standing CNTs grown on a silicon substrate demonstrating that the diameter and length of single CNT are quality uniform.

2.3 Characterization

The surface morphologies of a silicon pillar array were examined using a JEOL-6550 high resolution FE scanning-electron microscope (FE-SEM) with x-ray energy dispersive spectroscopy capabilities. To evaluate the performance of the individual CNT probe, this paper measured the surface morphology with the probe by a commercial AFM system (Veeco CP-II). The CNT probe is equipped with a silicon handle substrate. The dimension is compatible with the probe holders of almost all the commercial AFM instruments.
3. Results and discussion
A high resolution TEM image as shown in figure 4 illustrated CNTs are multi-walled hollow tubes. Figure 5 shows the SEM micrograph of a single CNT grown on the front-end of the silicon nitride cantilever which was used for surface characterization described in section 2.3.

Figure 6 shows the surface morphologies of a silicon pillar array with 1 µm spacing imaged by the single CNT probe. The measurement parameters are 8 µm×8 µm scan size and in a contact mode with 0.1 nN set-point force. Comparing the images in figure 6, the individual CNT probe can measure much more details of the sample than commercial silicon based AFM probes.

![Figure 6](image)

**Figure 6.** The AFM image of a standard sample by the CNT probe. The measurement parameters are 8 µm×8 µm scan size and 0.1 nN set-point force.

4. Conclusion
This paper developed a novel micro-cantilever equipped with a high aspect ratio CNT tip to perform surface characterization with high spatial resolution. The experiment results show that the E-beam lithography technique is capable of defining a nano-sized catalyst on the micro-cantilever array. A single vertical CNT was then synthesized by using an ICP-CVD system. The developed probe of this paper has demonstrated an ultra fine resolution in a contact mode and 0.1 nN set-point force without wearing out the probe and sample.

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6. Reference
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