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A quantitative comparison of resolution, scanning speed and lifetime behavior of CVD grown Single Wall Carbon Nanotubes and silicon SPM probes using spectral methods

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Abstract. Due to their extreme aspect ratios and exceptional mechanical properties Carbon Nanotubes terminated silicon probes have proven to be the “ideal” probe for Atomic Force Microscopy. But especially for the manufacturing and use of Single Walled Carbon Nanotubes there are serious problems, which have not been solved until today. Here, Single and Double Wall Carbon Nanotubes, batch processed and used as deposited by Chemical Vapor Deposition without any postprocessing, are compared to standard and high resolution silicon probes concerning resolution, scanning speed and lifetime behavior.

1. Introduction

Since the first demonstration of Carbon Nanotubes (CNTs) terminated silicon tips for their use in Atomic Force Microscopy (AFM) [1], CNTs have proven to be the “ideal” probe. Firstly, due to their extreme aspect ratios, its diameters in the nanometer range with lengths up to several microns, CNTs tips are coming close to the theoretically perfect scanning probe, i.e. a delta peak shaped profile. Secondly, the single molecule structure of the CNTs and the exceptional mechanical properties leads to unsurpassing stability, flexibility and low wear-out behavior. Manufacturing and state-of-the-art use of these CNT-probes splits up mainly into two fields: Multiwalled Carbon Nanotubes (MWNTs) are mostly attached manually to the silicon tip [1, 2]. MWNTs whose diameters are too large (i.e. above 10nm) for high resolution measurements. Direct growth of CNTs at the tip apex by Chemical Vapor Deposition (CVD) [3, 4] is leading mostly to CNTs with single (SWNTs) or very few walls. Due to their small radius SWNTs are considered to be best suited probes for high resolution measurements [4-6]. The technological issue for growing SWNTs is the deposition process, which must lead to a sole CNT at the tip apex. Also, the non-linear dynamic of SWNTs during scanning could lead to scanning artefacts [7-9]. A method to overcome the bending problem is the shortening of individual CNT tips by electrical pulses [10], an extensive and time-consuming process. Unfortunately until today there are no batch processed, directly useable SWNT AFM probes available.

Here, we present a detailed comparison of AFM measurements of standard and high resolution silicon tips together with batch processed, as deposited CVD grown Single or Double Wall CNT terminated tips on samples with regular structures in the nanometer regime. To quantify the gain of resolution using our CNT tips, we use a spectral analysis involving measurement of roughness power spectral densities.

2. Methods

Isolated Single or Double Wall CNTs are grown in a batch process at the apex of commercially available silicon SPM probes (Nanosensors™ Pointprobe® Plus) by the method of Hot Filament CVD (HFCVD) [11, 12]. The CNTs are used for AFM imaging as deposited during HFCVD. The sticking of the CNTs at the tip apex is very good. No additional fixture is applied. The lenghts of CNTs can be controlled up to a certain limit. The resulting CNT lenghts are suitable for the use in the AFM. No shortening or other postprocessing procedure is performed. Figure 1 shows an as-deposited Single or Double Wall CNT terminated silicon AFM probe.

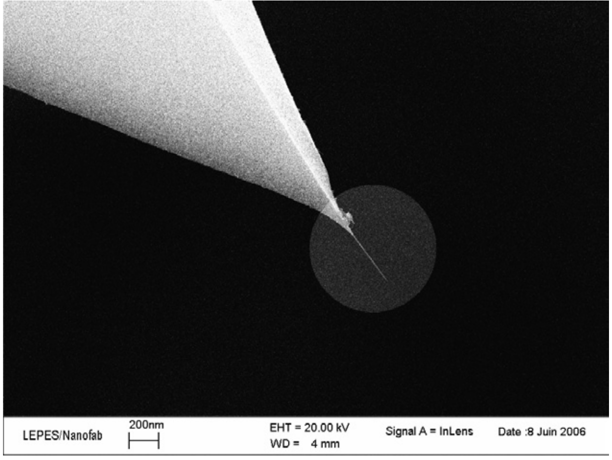

Figure 1: SEM image of an as-deposited Carbon Nanotube.

The CNT terminated probes are compared to standard silicon probes (Nanosensors™ Pointprobe® Plus, mean tip radius 7nm) and high resolution silicon probes (Nanosensors™ SuperSharpSilicon™ (SSS), mean tip radius 2-3nm) all with NCH-type cantilevers (nominal force constants 42N/m, nominal resonance frequencies 330kHz). All AFM measurements were performed either on a Veeco DI 3100 AFM with a Nanoscope V controller or a Veeco DI 5000 AFM with a Nanoscope IIIa controller. All images were taken under intermediate tapping conditions (free amplitude about 30nm, relative setpoint 0.6) in ambient atmosphere, pairwise on the same AFM. To quantify the gain of resolution Power Spectral Density (PSD) analysis is performed. PSD spectra are obtained from Fast Fourier Transformation of horizontal scan lines. The analysis of PSD spectra gives a quantitative and comparable value for each wavelength of spatial distribution [13-15].

3. Results

3.1. High resolution measurements on nanoporous silicon

Figure 2 shows images of nanoporous silicon measured with a CNT and a SSS tip, respectively. The CNT can resolve the fine structure of the nanoporous silicon much better than the SSS tip, which is clearly visible from the section in figure 3a. This is resulting in higher PSD signals at all wavelengths (figure 3b). For wavelengths of 1nm the CNT tip has 1.7 times, for 10nm wavelengths a 6 times higher intensity compared to the SSS tip. This is a tremendous increase in resolution compared to the most advanced commercially available silicon probes.

3.2. High resolution - high speed measurements on 2D100 calibration standard

A Nanosensors™ 2D100 calibration standard (inverted pyramids, edge lenghts about 50nm, pitch about 100nm etched into <100>-orientated silicon) was imaged with a CNT, a SSS and a standard silicon tip (figures 4a-c). The bottom edges of those pyramids, which are almost atomically sharp, could be reached the better the sharper the probe is. From the sections picture (figure 5), it can be seen

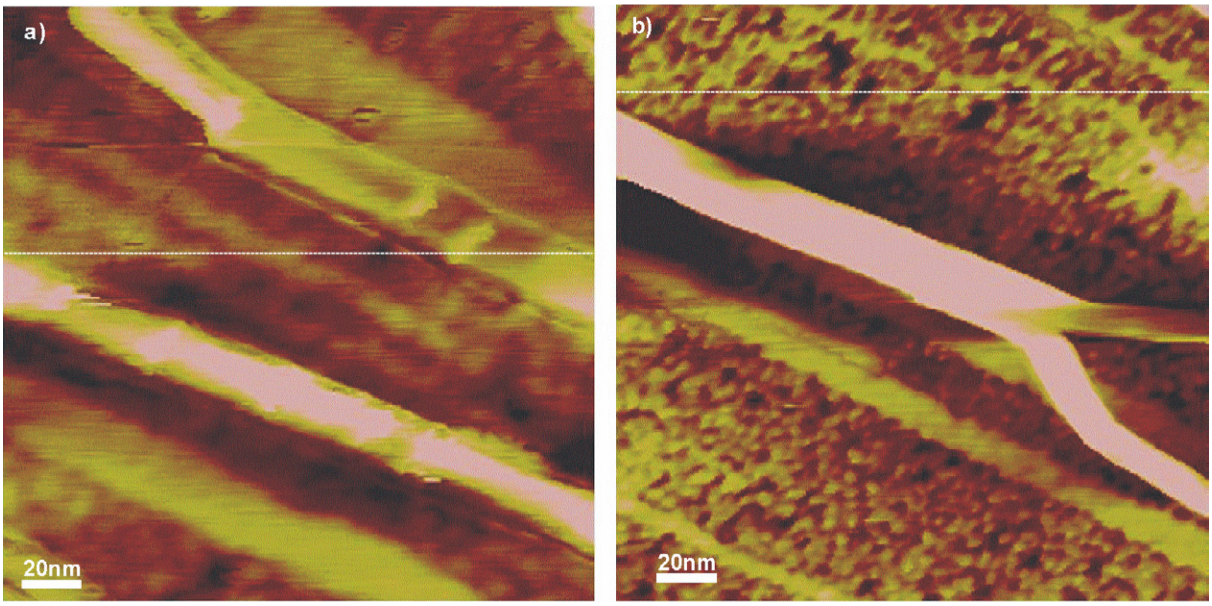

Figure 2: Scan of silicon nanopores measured a) with a SuperSharpSilicon™ and b) with a CNT probe. Scanning speed 1µm/sec. Height scale 5nm. The dashed lines refer to the places where the section plots in figure 3a were taken from.

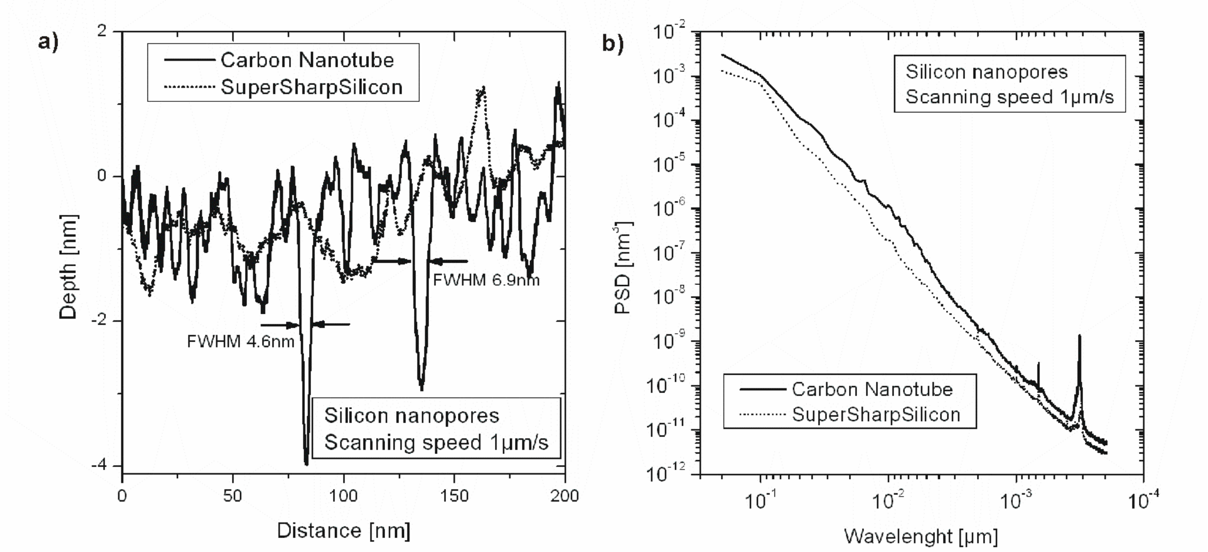

Figure 3: Section (a) and PSD spectra (b) of scans from figure 2 of using either CNT or SuperSharpSilicon™ tips on nanoporous silicon.

clearly that the CNT tip can penetrate the pyramids deeper and image the bottom most reliable. The highest resolution while using the CNT tips is resulting in the highest PSD values for wavelengths smaller than 10nm. Additionally, it can be seen from the PSD plot that the 10µm/sec CNT tip high speed scan has the same resolution than the 1µm/sec SSS tip scan. An increase of scanning speed by a factor of 10 is possible without obtaining less resolution than with the SSS probe.

3.3. Lifetime behavior - wear-out measurements on polycristalline silicon

Polycristalline silicon was imaged with a SSS and a CNT tip. The first image was taken with a scanning speed of 1µm/sec ("before aging"). Then, 100 images were taken with a speed of 10µm/sec ("aging procedure"). Afterwards, one image was taken again with 1µm/sec ("after aging"). From the

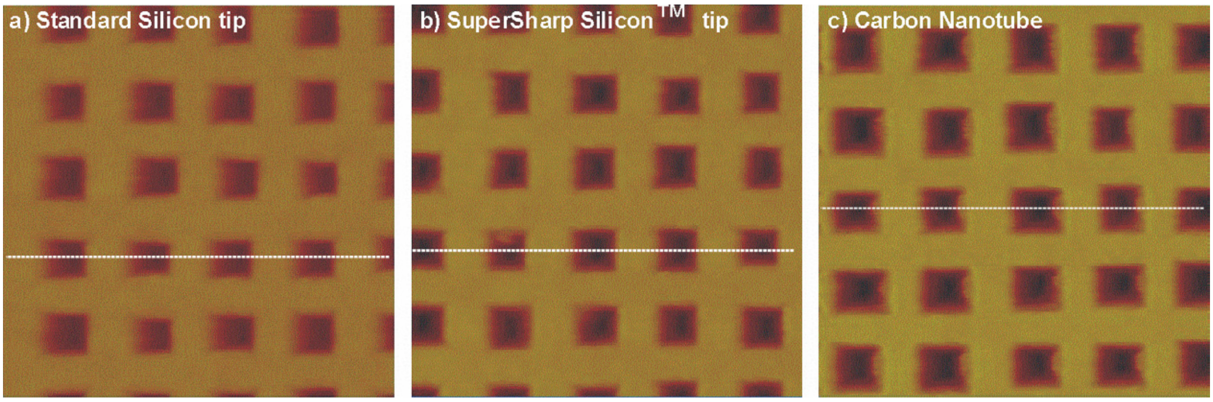

Figure 4: AFM images of Nanosensors™ 2D100 calibration standard. Imaged with a) a standard silicon tip, b) a SuperSharpSilicon™ tip and c) a Carbon Nanotube. The image sizes are 512nm by 512nm. Height scale 50nm. Scanning speed 10µm/sec. The dashed lines refer to the places where the section plots in figure 5a were taken from.

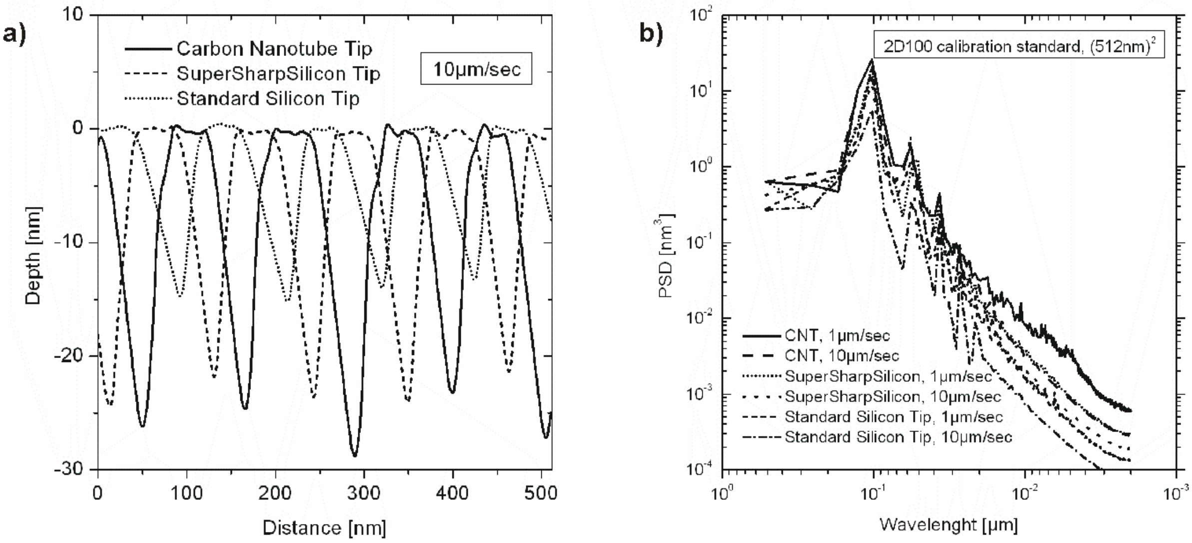

Figure 5: a) Section through the deepest parts of the pyramids of Nanosensors™ 2D100 calibration standard from scan of figures 4a-c. Scanning speed 10µm/sec. b) PSD spectra of standard silicon, SuperSharpSilicon™ and CNT tips on 2D100 calibration standard. Scanning speed is 1 and 10µm/sec.

PSD spectra in figure 6 no significant degradation of PSD signal of SSS and CNT tips due to the aging procedure could be observed. For both measurements, the CNT PSD signal for wavelengths below 20nm is increasing constantly compared to the SSS PSD signal denoting a higher resolution. For a wavelength of 4nm the CNT has a three times higher PSD signal than the SSS. This is related to a much better resolution of smallest details.

For wavelenghts much larger than the tip diameters (20-50nm) the PSD spectra is showing a different behavior compared to figure 3b. Both tips can track the surface of the sphere-like poly silicon properly. Only the very narrow gaps between two spheres are imaged more accurately by the CNT tip (same PSD values for 20-50nm, higher CNT PSD values for 1-10nm). Instead of that, the holes of the nanoporous silicon in 3.1 can not be penetrated as good with the pyramidal-shaped SSS tip than with the cylindrical-shaped CNT tip. Due to the smoothing of the surface image by the SSS tip pyramid, the PSD intensity for all wavelenghts is decreased compared to the CNT PSD signal.

For wavelengths below 2.5nm all PSD signals raise remarkably. This increase is caused by scanning artifacts due to feedback loop oscillations whilst scanning over upward steps.

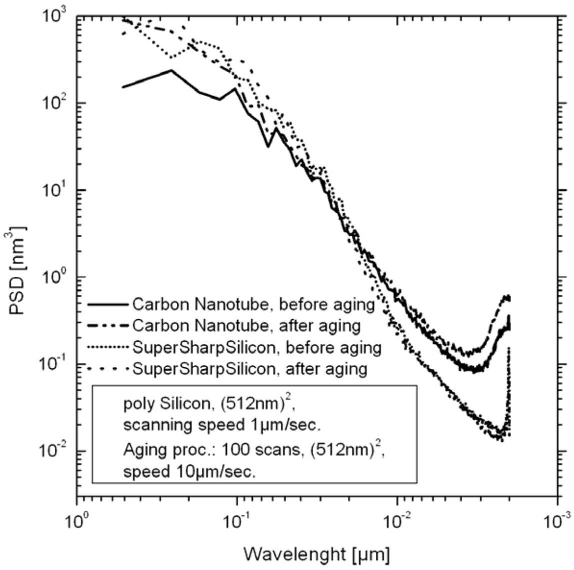

Figure 6: PSD spectra of SuperSharpSilicon™ and CNT tips on poly silicon. Before and after an aging procedure (100 scans, size 512nm by 512nm, scanning speed 10μm/sec.).

4. Conclusion

Batch processed Single and Double Wall Carbon Nanotube tips have been compared to standard and high resolution silicon probes concerning resolution, high speed behavior and lifetime. The Carbon Nanotubes are used as deposited during HFCVD in AFM. No postprocessing or CNT shortening is necessary. It has been shown quantitatively that the CNT tips could resolve sub-10nm details far better than high resolution silicon tips. Also, the CNT probes were used with a ten times higher scanning speed than high resolution silicon probes still acquiring images with the same resolution. No wear-out effects of CNT tips could be detected during long-term measurements.

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