Application Note:
Growth Uniformity Measurement for VAMWCNTs on 4" Wafer

Summary:
A method has been developed to measure the height uniformity of larger size, vertically aligned, multi wall Carbon Nano Tubes arrays grown on flat substrates. This method has been applied, to measure the typical height uniformity of a typical VAMWCNT Array grown on a 4" round Si wafer with ET3000-CNT system manufacturing by First Nano. A relative standard deviation of less than 3% was found across the whole 4" wafer. No special process optimization was done to obtain this test sample.

Introduction:
Carbon Nanotubes (CNTs) are promising candidates for many novel applications. One of the holdups in their commercialization is the lack of information how cost effectively they can be scaled up to commercial volume. Chemical vapor deposition (CVD) are used among other processes to produce single wall (SW) and multi wall (MW) CNTs with high quality and purity on Si wafer and other substrates and on larger size substrates. The uniformity of the CNTs yield on larger size substrates is one of the key factors enabling future commercialization scale-ups.
In this Application Note, a MWCNTs height uniformity measurement procedure is presented and applied to a typical mm tall VAMWCNT array grown on a full size 4" wafer to test the growth uniformity of an Easy Tube 3000 system with a 5" processing tube. Data obtained with this procedure can be used to further optimize the uniformity of the CNT growth across larger size wafers.

CNT growth sample preparation

1. Thin film catalyst and substrate preparation

We used an e-beam system and a thermal oxidation process to deposit typically a three layer catalyst multi stack on a 4" Si wafer (001). First we oxidized the Si wafer in an ET3000 overnight to obtain a 500 nm thick SiO_2 layer. Then we e-beam deposited an Al thin film layer, typically 10 nm thick, on top of the SiO2 layer and subsequently oxidize it in a thermal oxidation process. Then we e-beam deposit another 0.5-2nm layer of iron, (typical 1 nm) to complete the catalyst wafers manufacturing process.

2. VAMWCNT array growth on Si wafer

An ET3000 system (FN0004 (as shown in Figure 1) located in our First Nano Application Laboratory,) was used to grow a mm tall VACNT array on a 4" Si wafer to obtain a test sample for the below describe procedure. Typically we used methane as carbon precursor material, but other carbon containing gases can be used as well. The thermal CVD process used a process temperature of 400-1000C depending on the substrate material used and types of CNT needed. To make VACNT forests we typically 750C and methane as carbon precursor gas.

Sample Preparation for growth uniformity test

After growing the VAMWCNT array on a Si wafer, the Si wafer has been scribed and broken in half and pulled apart as shown in Figure 2. A secondary scribe has been used to narrow the sample further so that is could better fit the SEM sample holder (we kept the edge from the first cut untouched)

Sample Measurement

After the sample was then mounted to the SEM sample holder and loaded into SEM. Working distance, contrast, brightness, and magnification, etc should be adjusted for measurement and images should then be taken. The representing SEM images has been shown in Figure 3 a and b.

Record the position of the wafer edge as the reference point. Move the sample along the horizontal direction and record MWCNT growth starting position. Record other positions along the split line for every 2 to 5 mms, followed by MWCNT growth ending position and finally the other edge of the silicon wafer. Whenever there is a curve on the profile, more points should be recorded to fully capture the curve, as shown in Figure 3c-f. SEM images had been taken for each position recorded.

Results

The sample has been measured at 36 different locations across the wafer using SEM. The data points are been shown in Figure 4, all of which have been used to derive the average. Table 1 lists statistic data for the measured CNT heights.

Figure 1: EasyTube 3000 system

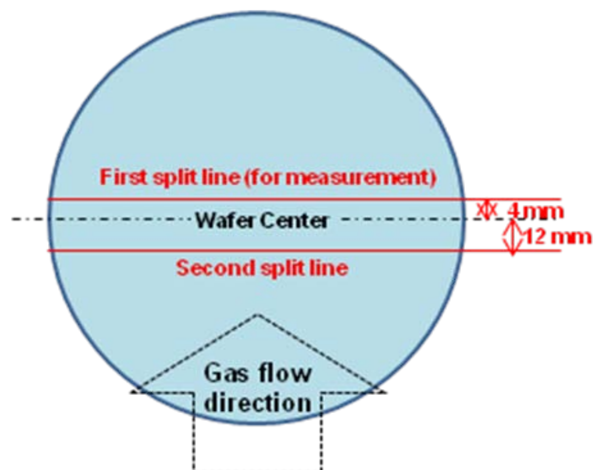
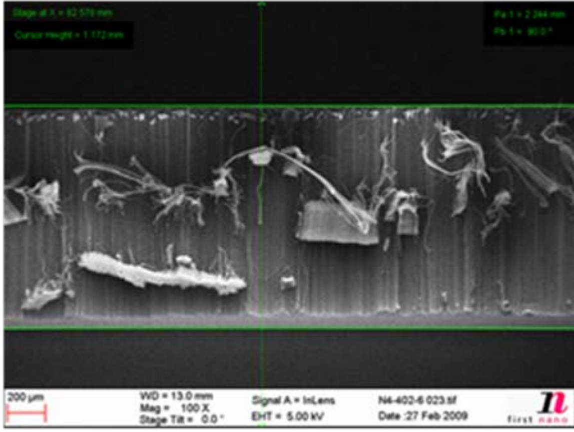
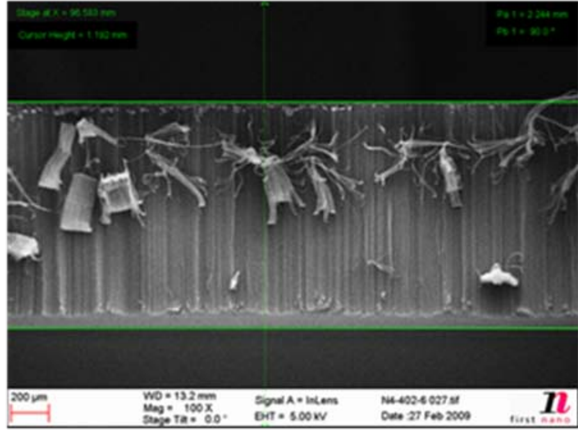


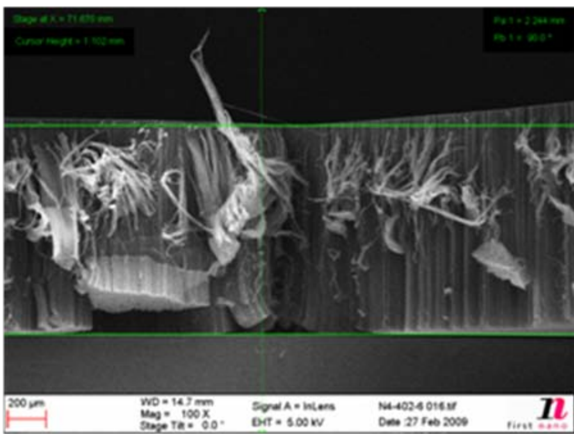
Figure 2: Split sample for SEM measurement



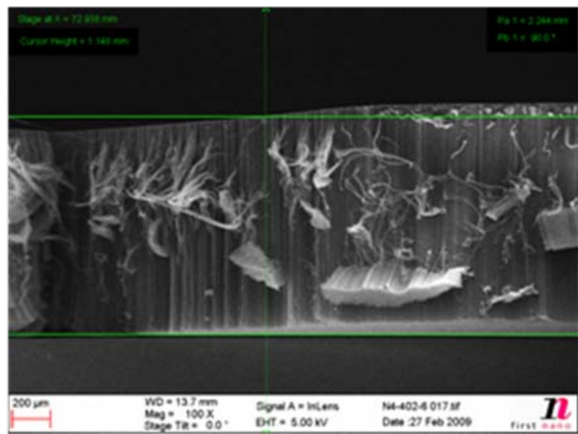
(a)



(b)



(c)



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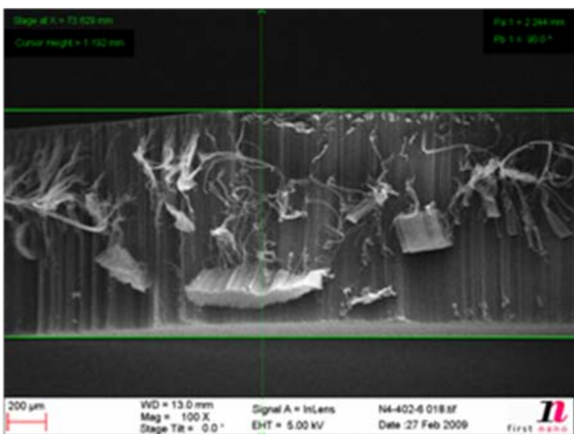
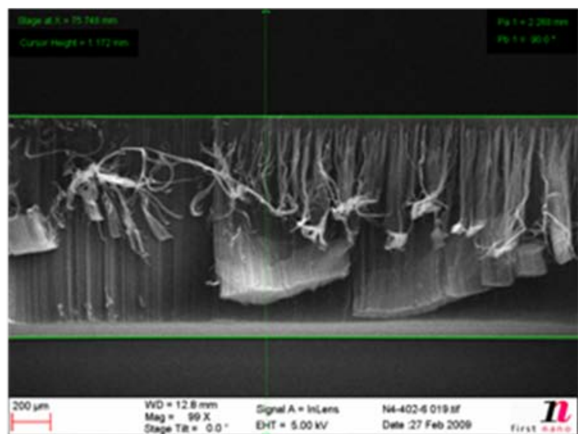


(e)



(f)

Figure 3: SEM images of CNT film over different spot from the same sample

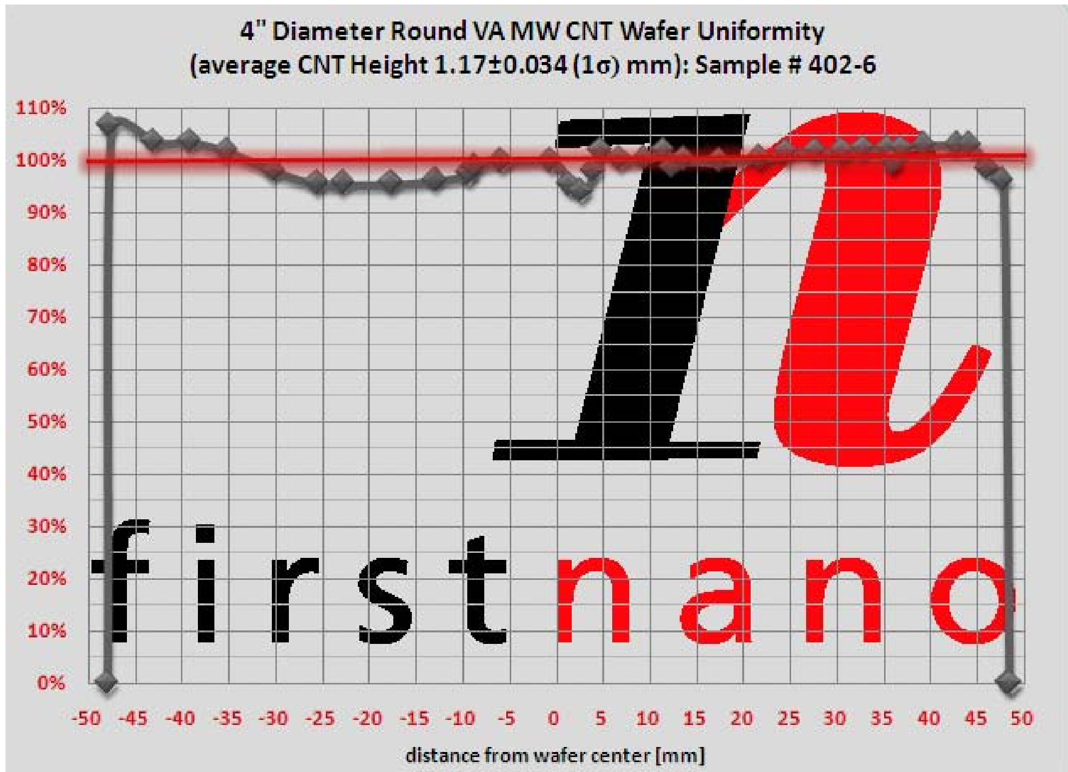


Figure 4: MWCNT wafer uniformaity

Table 1: CNT height Data

Sample Number 402-6	Height	Percentage
Average Height	1171 um	100%
Maximum height	1248 um	107%
Minimum height	1102 um	94%
Standard Deviation	34 um	2.9%