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Maximizing Carbon Nanotube Forest Growth with 99.5% Pure Ethylene

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ABSTRACT

A new growth recipe for height maximization of carbon nanotubes (CNTs) by thermal chemical vapor deposition is found using an ethylene purity of 99.5%. This new recipe yields fast growth-rates with average growth heights of 1146 µm in 10 minutes of growth. The CNT forest growth is very uniform but also has tears in structures, bowed vertical growth, and rough sidewalls indicating a poor quality CNT forest. These poor features seem to be a result of the thickness of the iron catalyst layer. With a thicker catalyst layer, high quality CNT forests may potentially be grown using 99.5% pure ethylene with faster growth rates than the current standard growth recipe for CNTs using 99.95% pure ethylene.

INTRODUCTION

Carbon nanotubes (CNTs) are cylindrical fullerenes with properties which make them widely researched and ideal for applications in electronics and micro fabrication. CNTs can exist as either single-wall nanotubes (SWNTs) or multi-wall (MWNTs) nanotubes with free standing structures of diameters as small as half a nanometer[1]. They are often grown with vertical alignments into thick forests of many CNTs which are held together by Van der Waals forces for the purposes of easier fabrication and manipulation[2].

CNTs have the greatest length to diameter ratio of any current known material with ratios up to 28,000,000:1[3]. In addition to this, they also possess the greatest strength and elastic modulus of any fabricated material[1]. CNTs have useful electrical properties with excellent electrical conduction and can handle current densities up to a million times that of silicon[4-5]. They are also excellent thermal conductors and are extremely stable through temperature change[6]. These properties make them excellent candidates for use in fabrications of microelectromechanical systems (MEMS), electrical circuits, sensors, solar cells, and use in many other structural and electrical applications[7-9].

CNT forests were first grown in 1992 through an arc-discharge method[10]. A year later, Thermal Chemical Vapor Deposition (TCVD) was used to grow CNT forests and has become the most common method of CNT growth[11]. A few other methods of CNT growth have been discovered including laser ablation, but these methods have proven to be more expensive and less successful than that of arc-discharge and TCVD[12].

Several factors influence growth-rate and other factors of CNT forests when grown by TCVD. These include the use of a metallic catalyst, the thickness of this catalyst, the temperature at which growth occurs, and the hydrocarbon type and flow-rate. In all cases, the growth-rate decreases as the time of growth increases due to amorphous carbon encapsulating catalyst particles[13].

The use of a metallic catalyst stimulates growth of carbon nanotube forests onto a substrate. Commonly used catalysts include Nickel, Iron, and Cobalt. Huang et al. discovered that of these catalysts, Nickel produces the greatest growth-rate, largest diameter of CNTs, and the smoothest sidewalls. A cobalt catalyst gives the slowest growth-rate, smallest diameter, and roughest sidewalls and Iron catalysts yield results between nickel and cobalt. Although CNTs grown with a Nickel catalyst provide the fastest growth-rate and smoothest sidewalls, the large diameter of each CNT decreases the strength of the forest[14].

The thickness of the catalyst determines if SWNTs or MWNTs are grown. It also affects the growth-rate and forest density[15]. Faster growth-rates and thicker forest densities are achieved by decreasing the thickness of the catalyst; however, when catalyst thickness decreases below 1 nm, growth becomes unstable[15-16].

The temperature at which CNT growth occurs affects the forest growth-rate and individual tube diameter. Growth-rates increase with temperature as does the diameter of CNTs. Additionally, increased temperatures can cause CNTs to compartmentalize into short CNTs stacked on top of each other, creating rougher walls[17].

The type of hydrocarbon and flow-rate of hydrocarbon gas also impacts the growth-rate and quality of CNT forest growth. Different hydrocarbons will produce maximum growth rates when placed in different growth conditions and with different flow-rates. The flow-rate to maximize growth rates in comparison to the flow-rate of hydrogen gas during growth also changes depending on the type of hydrocarbon gas being used[18].

The combination of all of these factors in CNT forest growth can be combined into an optimal growth recipe which yields predictable growth-rates and features. For the purposes of this report, the optimal growth recipe means the recipe yielding the tallest CNT structures without sacrificing uniformity of forest height throughout a structure. When the purity of hydrocarbon gas being used changes, the recipe to maximize growth-rates while keeping desirable features such as uniformity of growth, straightness of growth, and sidewall roughness will produce different results; therefore, a new recipe must be determined. A factorial study was performed to find the optimal growth recipe for CNTs when the Ethylene purity was reduced from 99.95% to 99.5%.

METHODS

Preliminary tests by Jun Song (personal communication, March 30, 2009) and previous studies by Brendan Turner were used to determine which factors should be tested and provided the initial average values for each factor[16]. The thickness of the iron catalyst, hydrogen gas flow-rate, and ethylene gas flow-rate were chosen as factors to study as they seemed to have the greatest affect on CNT forest growth-rate and features. Table 1 gives the assigned low, average, and high values initially chosen for the experiment.

	Low	**Average**	**High**
Iron Catalyst Thickness	≈0.3 nm	≈0.4 nm	≈0.5 nm
Hydrogen Flow-rate	190 sccm	280 sccm	378 sccm
Ethylene Flow-rate	59 sccm	118 sccm	174 sccm

Table 1 Experimental factors and assigned values.

There are a total of nine combinations of growth recipes from the above factors, which were to be repeated three times for a total of 27 runs. All other factors were held constant throughout the experiment including a growth temperature of 770° C, a growth time of 10 minutes, and an alumina layer thickness of approximately 30 nm. Those factors were chosen based upon success with former CNT growths. The growths were completed in random order and preceded by an air clean of the same growth system. A pattern emerged after the first trial in which all growths with a hydrogen flow-rate of 378 sccm yielded significantly slower growth-rates and non-uniform growth. These four runs were removed from the second and third trials.

FABRICATION

Sample fabrication included several steps which can be grouped into sample preparation and CNT growth. The steps of sample preparation include deposition of an alumina layer on a silicon wafer, patterning by photolithography, iron catalyst deposition, and lift-off of photo resist. The steps of growth include annealing the iron catalyst layer and flowing ethylene simultaneously with hydrogen gas at the predetermined growth temperature.

A 30 nm layer of alumina was deposited onto 4 silicon wafers with an E-beam evaporator. The alumina layer is necessary for CNT growth because it keeps the iron catalyst from agglomerating and diffusing into the silicon wafer when it is heated up to growth temperature[19]. After alumina deposition, each wafer underwent a simple clean which involves a 30 second acetone rinse and 1 minute acetone bath is quickly followed by a 30 second IPA rinse and 1 minute IPA bath. Wafers were then thoroughly dried with nitrogen.

After wafers were cleaned a positive photo resist, AZ-3330, was spun at 5500 rpm for 60 seconds to approximately 2 µm and soft baked at 90 °C for 60 seconds. Wafers were then exposed under a patterned mask for 10 seconds and developed in AZ-300 MIF for 35 seconds followed by a rinse in deionized water, drying with nitrogen, and post bake at 90 °C for 60 seconds. Although the developer etches alumina, the exposure for 35 seconds does not etch enough alumina to affect CNT growth[7].

When patterning by photolithography was completed, iron was deposited onto the wafers with a thermal evaporator. Iron was deposited on three wafers at a rate according to the crystal monitor to be 1.0 Å/sec. for 3 seconds for the first wafer, 4 seconds for the second wafer, and 5 seconds for the third wafer as timed by a stopwatch. The samples then underwent a lift-off process as they were sonicated in a resist stripper, Microposit 1165, for 15 minutes and were cleaned by the same cleaning procedure as was used before adding and spinning photo resist.

The iron catalyst thickness of each sample was then measured in an AFM. The samples were measured to be 5.23 nm, 8.02 nm, and 11.80 nm respectively. These measurements were very different from the expected thicknesses of 0.3 nm, 0.4 nm, and 0.5 nm. Figures 1, 2, and 3 (ppg. 4-5) show the AFM images and height measurements of these samples.

It was later realized that the thicknesses measured in the AFM were not correct because of the method of fabrication. As the developer removed the exposed photo resist, it etched away at the alumina layer on the wafer. This in turn resulted in the iron layers appearing to be much thicker than they actually were. No witness sample was placed in the thermal evaporator as the iron was evaporated onto the wafers, so the exact iron layer thicknesses of the growth samples cannot be know. It was determined that a calibration of the deposition of iron with the same thermal evaporator would give accurate values of the approximate values of the actual iron thicknesses.

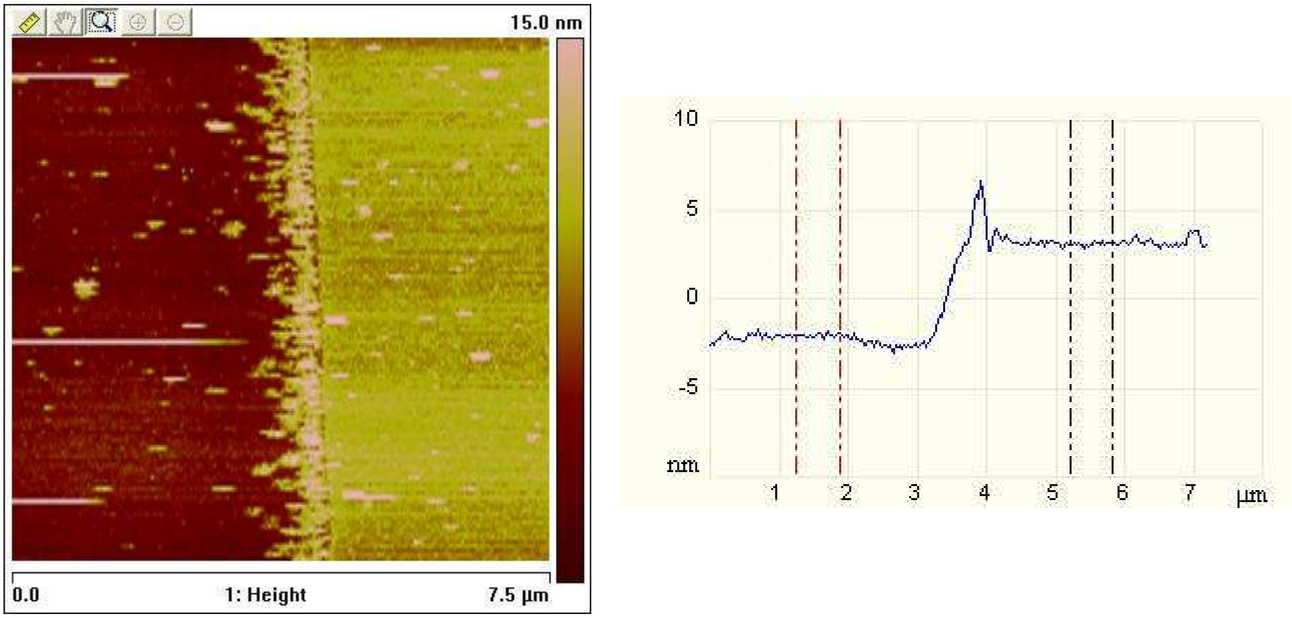

Figure 1 *left*: AFM picture of Silicon/Iron barrier. *right*: Measured height at 5.23 nm.

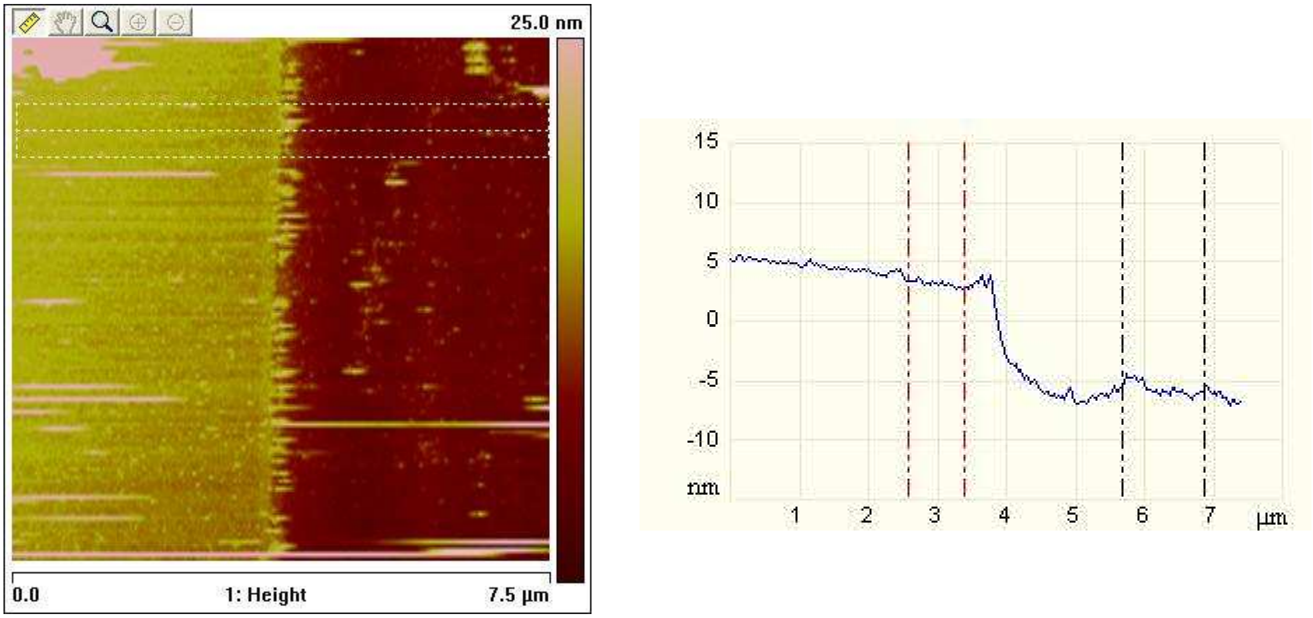

Figure 2 *left*: AFM picture of Iron/Silicon barrier. *right*: Measured height at 8.02 nm.

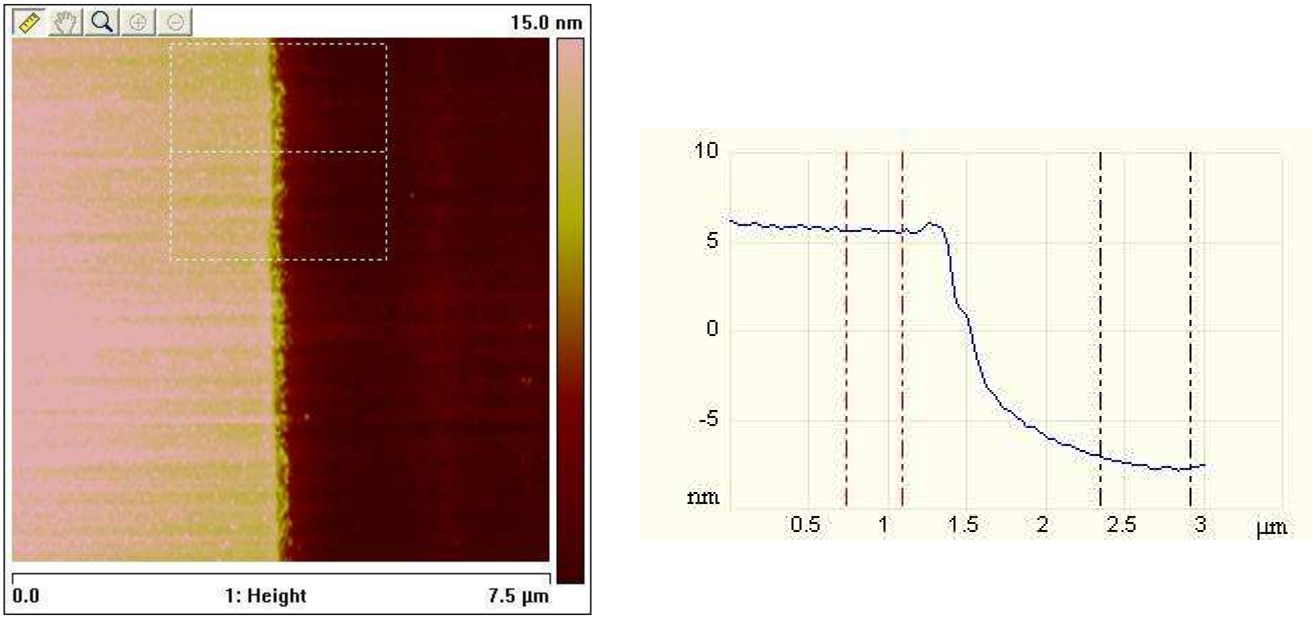

Figure 3 *left*: AFM picture of Iron/Silicon barrier. *right*: Measured height at 11.80 nm.

A calibration for the crystal monitor reading and actual iron thickness deposited on silicon wafers with alumina which had not received photo resist or been etched by AZ-300 MIF developer was performed by evaporating iron at a rate of 1.0 Å/sec. for several different lengths of time. An abruptor was placed over a portion of these wafers create a clean and straight edge of iron deposition which can easily be measured in an AFM[20]. The results of the calibration showed that the crystal monitor measures the iron deposited to be very similar to the actual thickness of the iron layer. Figure 4 gives the results of the calibration:

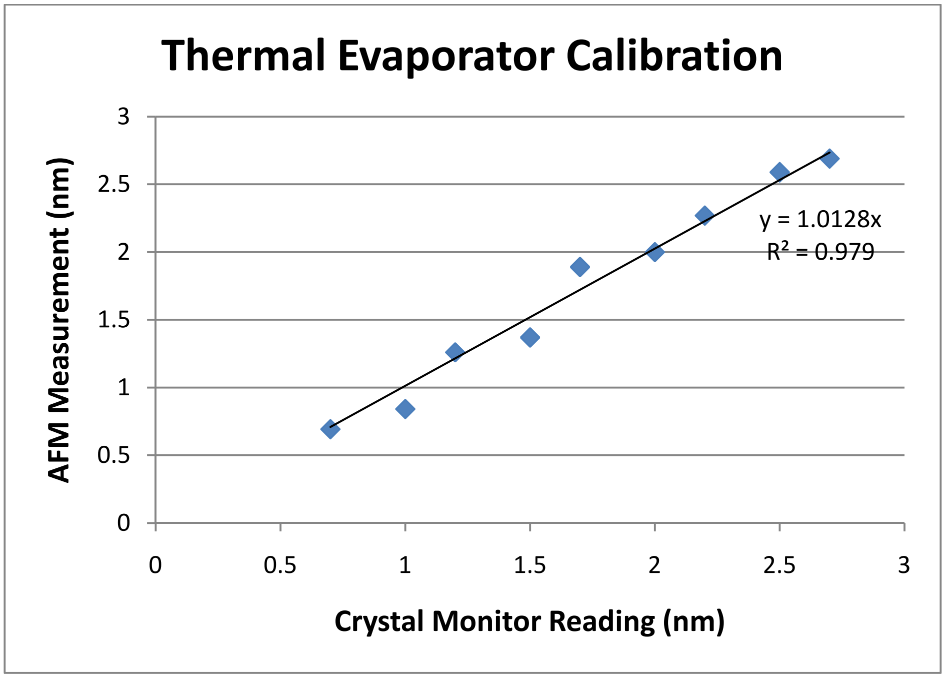

Figure 4: Table showing thicknesses measured by the AFM compared to readings by the crystal monitor for the thermal evaporator on silicon-alumina wafers not etched by developer.

A linear relationship exists between the actual thickness of deposited iron layers and the thickness measured by the crystal monitor. When the data is fit linearly with a fixed y-intercept of zero, a strong coefficient of determination of 0.979 exists. Using the linear relationship given in the plot above the actual iron thicknesses of the samples used to grow CNTs can be accurately estimated. The approximate iron thicknesses used for CNT growth are 0.304 nm, 0.405 nm, and 0.507 nm. These iron thickness layers are very close to the lower limit which will sustain CNT growth[21].

After iron thicknesses were measured, samples were annealed with hydrogen in a tube furnace up to 770 °C and then held at this temperature for 10 minutes of growth by TCVD with ethylene and hydrogen at the determined flow-rate for each run. The flow-rate of the gasses was controlled by mass flow controllers (MFCs). After 10 minutes expired the sample was cooled in the tube with argon. As previously discussed in the methods section each run was performed in random order with an air clean of the tube between each run.

Features of the CNT forests grown with 99.5% pure ethylene were then compared to features of CNT forests grown with 99.95% pure ethylene that were grown from the same fabricated wafer. Growing procedures were identical to the procedures listed above for the CNT forest growths with 99.95% pure ethylene except rather than repeating all trials, only the standard growth recipe for CNTs grown with 99.95% ethylene and the optimal, meaning tallest and most uniform, growth recipe found from the trials using 99.5% pure ethylene were completed for the comparison with CNT forests grown with 99.95% pure ethylene.

RESULTS AND DISCUSSION

Table 2 gives the data of this first trial; the growth heights were measured by SEM and the uniformity ranking is a qualitative ranking, with 1 being the most uniform growth, based on plain vision and SEM imaging.

	Fe Thickness (nm)	H_2 Flow-rate (sccm)	C_2H_4 Flow-rate (sccm)	Growth Height (µm)	Uniformity Ranking
*1	0.507	378	174	215	8
*2	0.507	378	59	155	7
3	0.507	190	174	1020	1
4	0.507	190	59	680	3
*5	0.304	378	174	219	9
*6	0.304	378	59	182	6
7	0.304	190	174	1040	2
8	0.304	190	59	576	5
9	0.405	284	118	679	4

Table 2 Trial 1 data from 10 minute growths at 770 °C using 99.5% pure ethylene.
*Runs in red were removed from further trials.

Upon completion of the first of three trials, a pattern was observed in which all growths with a high level hydrogen flow-rate (378 sccm) produced slower growth-rates and less-uniform growth than all other samples. This is most likely due to an excess of hydrogen gas etching the forests as they grow resulting in shorter growths and non-uniform growth. Subsequently, these runs were removed from trials 2 and 3.

The remaining trials were carried out while performing runs in random order. The growth heights were measured by SEM and optical microscope. Height measurements were given by the optical microscope by focusing on the top of a CNT forest of the sample and then measuring the number of microns the microscope needed to be lowered to focus on the silicon substrate. Uniformity rankings were assigned the same way as the first trial. After all data was collected, growth heights and uniformity rankings were averaged and the standard deviation in CNT forest heights was calculated as can be seen in Table 3.

	Fe Thickness (nm)	H_2 Flow-rate (sccm)	C_2H_4 Flow-rate (sccm)	Growth Height (µm)	Height Deviation (µm)	Uniformity Ranking
1	0.507	190	174	1073	45	1.33
2	0.507	190	59	735.7	56	4.33
3	0.304	190	174	1147	76	1.67
4	0.304	190	59	617	49	4.33
5	0.405	284	118	751.0	51	3.33

Table 3 Average growth heights, uniformity rankings, and the standard deviation of growth height.

Growths with an ethylene flow rate of 174 sccm produced the tallest and most uniform CNT forests in each trial. Growth was more uniform with an iron thickness of 0.507 nm for 2 of 3 trials, but the difference in uniformity between the 0.507 nm and 0.304 nm CNT forests with flow-rates of 190 sccm for hydrogen and 174 for ethylene were minimal. The standard deviation in CNT forest height was smallest for 0.507 nm thick iron with flow-rates of 190 sccm for hydrogen and 174 for ethylene while greatest for the 0.304 nm iron thickness samples with the same flow-rates. Although the standard deviation of growth height is greater in the 0.304 nm iron layer sample, its average growth height is large enough that it will still reliably grow to taller heights than the 0.507 nm iron layer samples. Because the runs with an ethylene flow-rate of 174 sccm were superior to runs with ethylene flow-rates of 59 sccm in terms of height and uniformity, they will be the only runs discussed throughout the duration of the results section and will be referred to by their iron thickness only.

In addition to these quantitative measurements SEM images provided information on the quality of the CNT forest growths. The features of growths of particular interest are the straightness of growth and sidewall roughness.

Straight growth is essential to most applications of CNTs. Straight growth will give greater stability to CNT forests, especially to thinner structures, making them essential for applications to MEMS. Figure 5 shows the sides of CNT structures for typical control (0.405 nm, 284 sccm H_2, 118 sccm C_2H_4), 0.507 nm iron layer, and 0.304 nm iron layer samples.

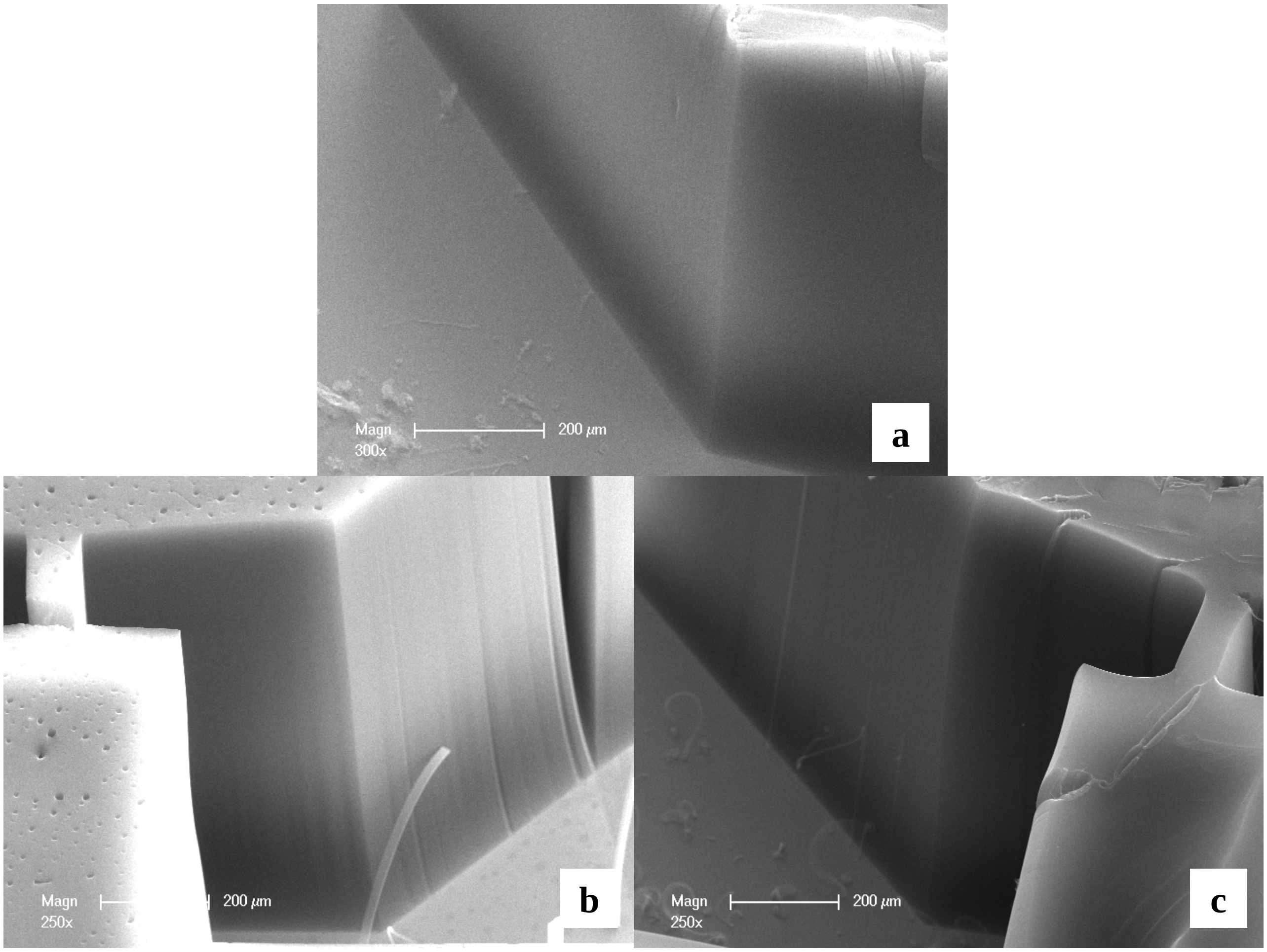

Figure 5 a) 796 µm tall CNT forest from a control sample showing slightly curved vertical growth. b) 1130 µm tall CNT forest from a 0.507 nm Fe sample showing significantly bowed-in vertical growth. c) 1210 µm tall CNT forest from a 0.304 nm Fe sample showing significantly bowed-out vertical growth.

Note: All images taken at a 50° tilt.

uniform 0.304 nm and 0.507 nm samples, which may be only because they are much shorter than the other samples. It is also interesting to note that although the 0.304 nm samples are taller, their growth is actually straighter than the 0.507 nm samples; this suggests that the 0.304 nm samples produce not only taller growth, but a better quality growth as well.

Sidewall roughness is a good indicator of the quality of CNT forest growth. Like straightness of growth, it becomes more significant with thinner structures and can determine if CNT forests can be useful as the geometrical frame for MEMS devices and other applications. Figure 6 shows the sidewalls of a control sample, a 0.507 nm iron layer sample, and a 0.304 nm iron layer sample.

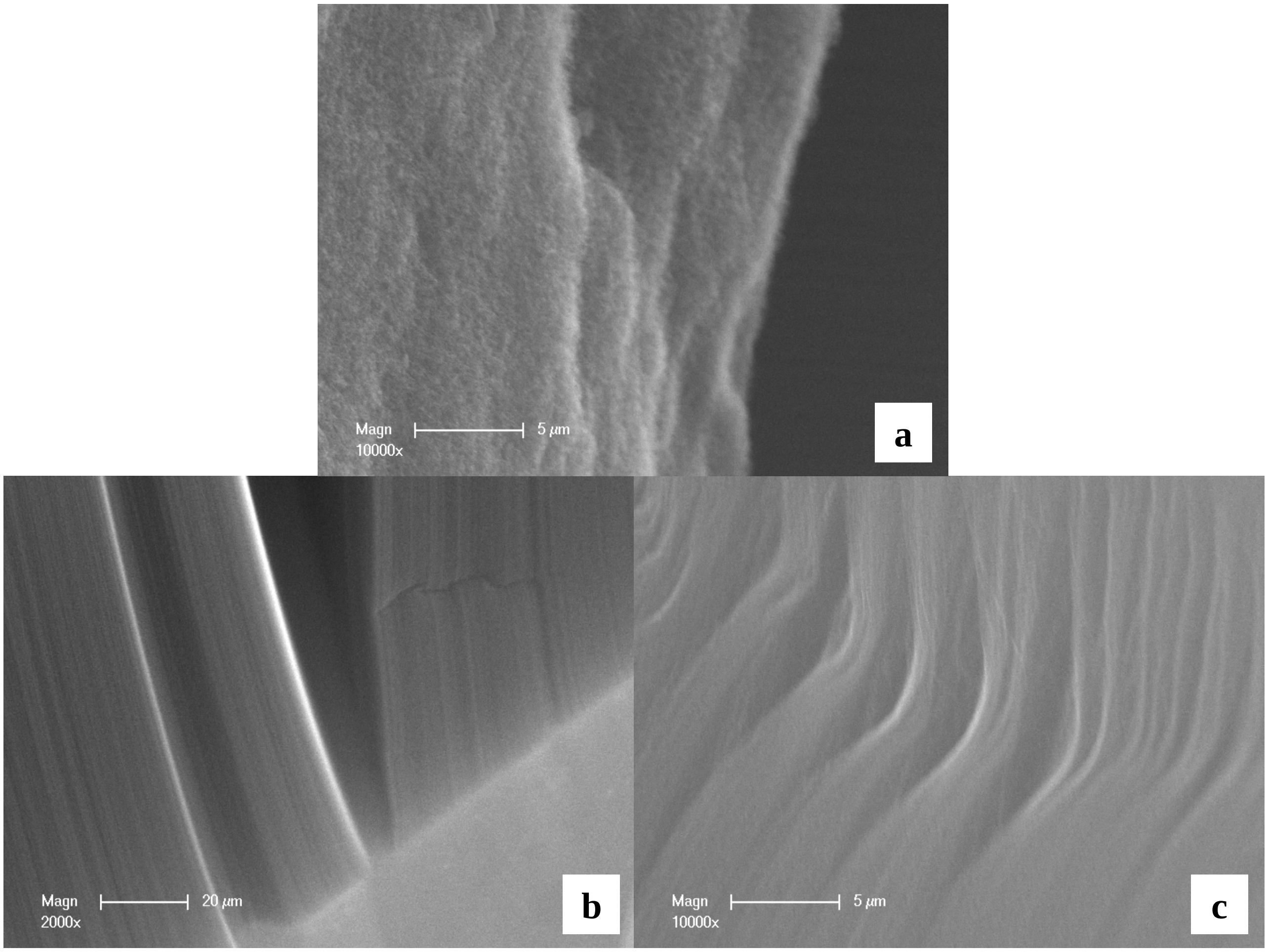

Figure 6 a) Control sample showing bumpy sidewalls at high magnification (10,000x). b) 0.507 nm Fe sample showing large divots and creases in sidewall at smaller magnification (2,000x). c) 0.304 nm Fe sample showing bumpy and curved sidewalls at high magnification (10,000x). *Note*: All images taken at a 50° tilt.

Figure 6 demonstrates that once again, CNT forest growth from these samples is not acceptable for most applications for CNTs. Once again, the control exhibited the most quality sidewall growth and the 0.304 nm iron layer sample was far better quality in sidewall features than the 0.507 nm iron layer sample.

In addition to straightness of growth and sidewall roughness, other interesting features were noticed in the forests of these samples. All samples seemed to have tears in the structures, which appears to be a result of non-straight growth. In addition to tears being present in the structures, the samples with an iron thickness of 0.507 nm had craters of varying sizes along all top surfaces. Figure 7 shows the undesirable features for a typical run of each sample.

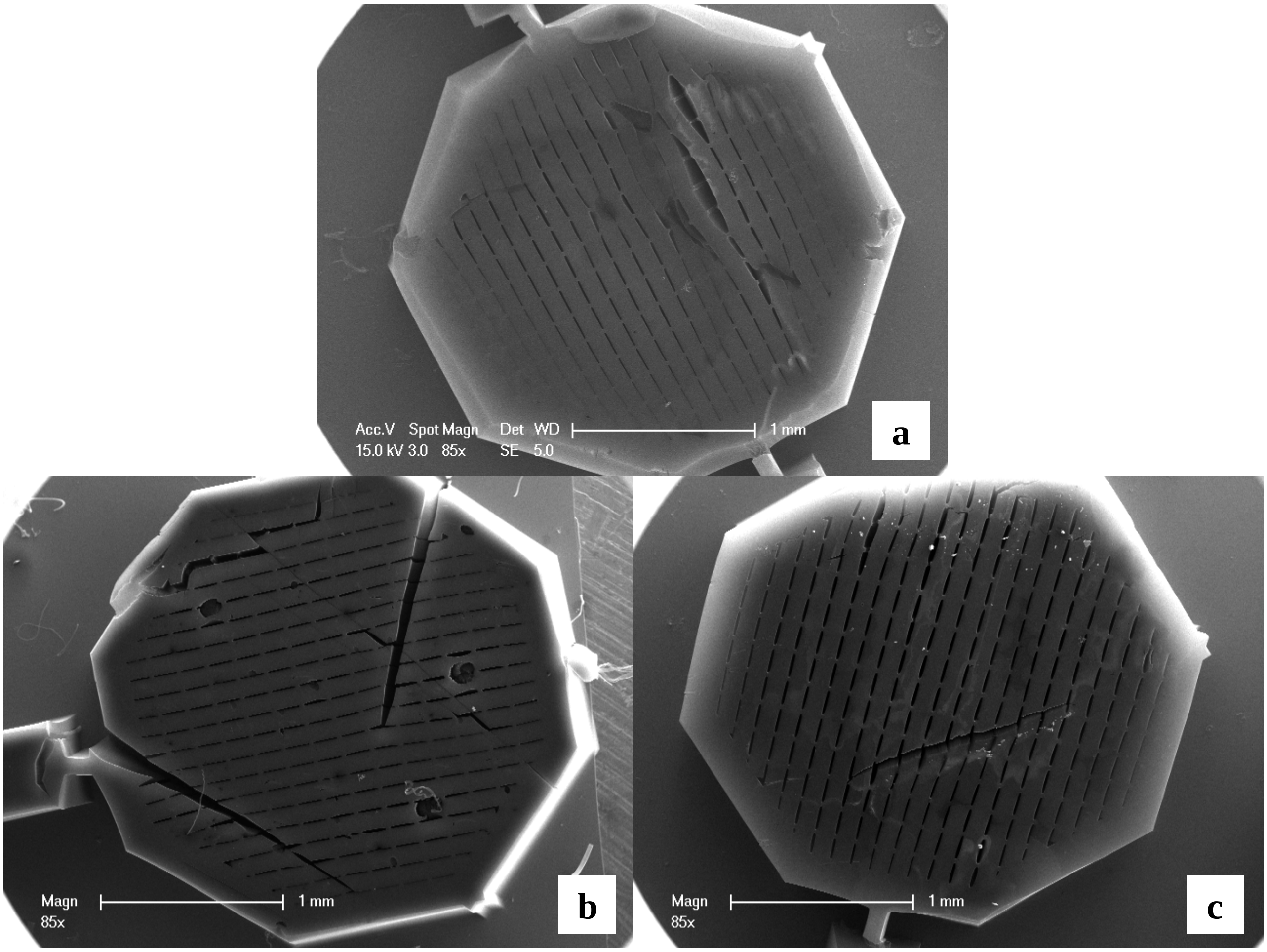

Figure 7 a) Control sample showing some tears parallel to patterned growth of structure. b) 0.507 nm Fe sample showing straight line tears in multiple directions on sample and craters of various sizes along surface. c) 0.304 nm Fe sample showing several tears moving traveling in multiple directions.

Tears were largest in taller structures which supports the theory that they are caused by non-straight growth. It also indicates that although CNT forests grown with 99.5% ethylene can be grown with fast growth-rates, the resulting undesirable features make the structures not particularly useful. As can be seen in figure 8, Craters formed in growth for runs with iron thicknesses of 0.507 nm were much cleaner with a lower flow-rate of ethylene which may be a result of less carbon present to grow CNTs.

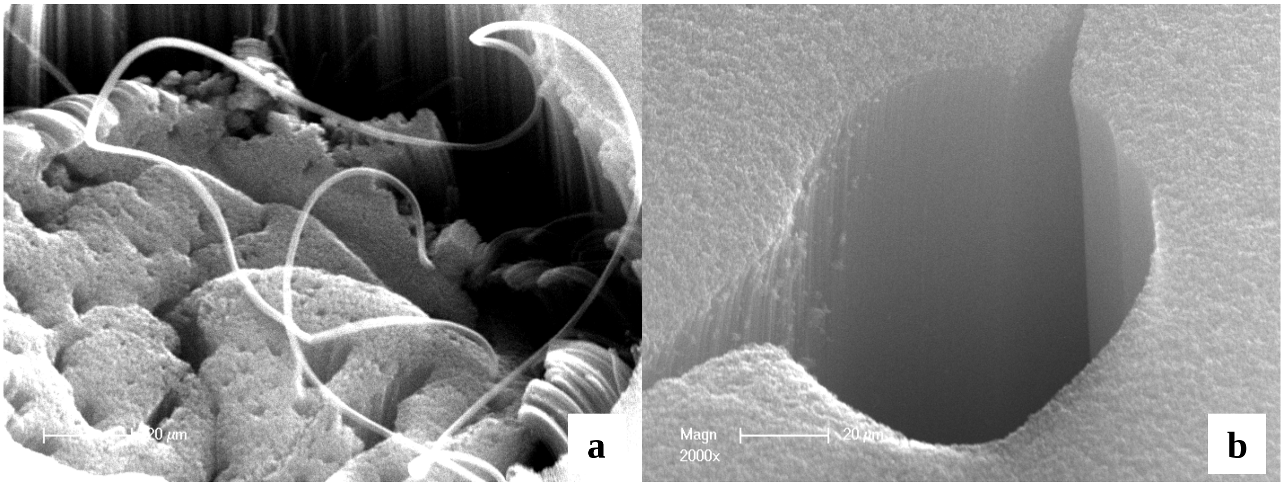

Figure 8 a) Very messy growth inside a surface crater on a 0.507 nm sample grown with 190 sccm H_2 and 174 sccm C_2H_4 taken at 2000x magnification. b) Cleaner growth inside a surface crater on a 0.507 nm sample grown with 190 sccm H_2 and 59 sccm C_2H_4 taken at 2000x magnification.

Note: All images taken at a 50° tilt.

The features of the CNT forests grown with 99.5% pure ethylene were then compared to the features of the CNT forests grown with 99.95% pure ethylene from the same fabricated wafer. The results of this comparison suggest that the undesirable features on CNT forests grown with 99.5% pure ethylene was not a result of the ethylene purity, but a result of the iron catalyst layer thickness. Figure 9 (page 12) shows non-straight growth, sidewall roughness, and tears in the CNT forests grown with 99.95% pure ethylene for the standard recipe and the new optimal growth recipe found for CNT growth with 99.5% pure ethylene.

Interestingly, the CNT forests grown with 99.95% pure ethylene had significantly shorter growth rates than CNT forests grown with 99.5% pure ethylene. Additionally, CNT forests grown with 99.95% pure ethylene had much greater growth rates when using the newly found optimal growth recipe for CNT growth using 99.5% pure ethylene then using the previous standard growth recipe for CNT growth. Growth with the standard recipe formed forests of about 346 µm in height and the new optimal recipe formed forests of about 772 µm in height; both measurements were made by SEM. These growth rates are much less than that of the forests grown with 99.5% pure ethylene.

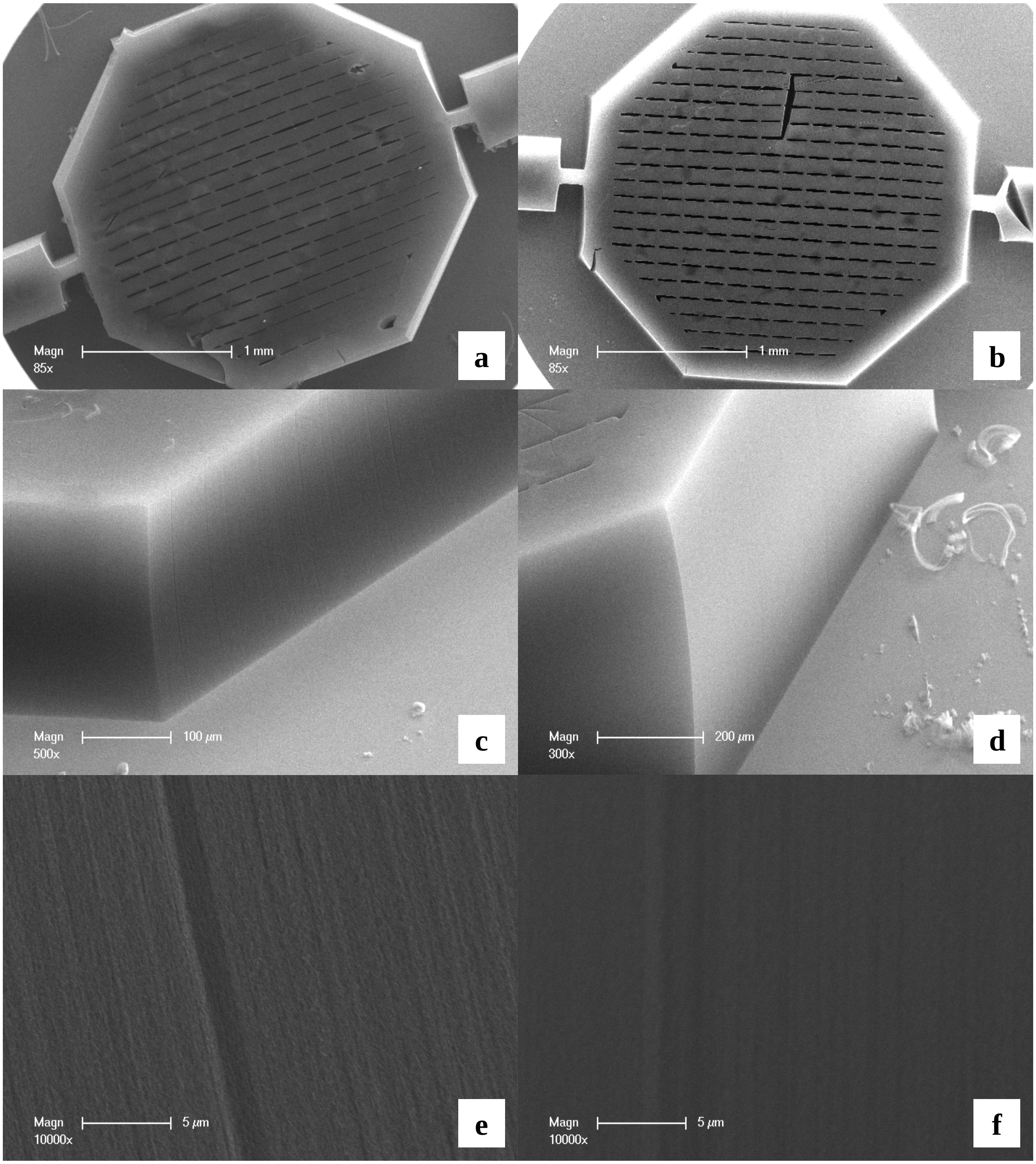

Figure 9 a) Standard growth recipe for CNTs grown with 99.95% pure ethylene showing thin tears in structure. b) Optimal growth recipe for CNTs grown with 99.5% pure ethylene using 99.95% pure ethylene showing larger tear on sample. c) Standard growth recipe for CNTs grown with 99.95% pure ethylene have significantly shorter growth and sidewalls bowed inward. d) Optimal growth recipe for CNTs grown with 99.5% pure ethylene using 99.95% pure ethylene showing shorter growth and sidewalls bowed outward. e) Standard growth recipe for CNTs grown with 99.95% pure ethylene showing some sidewall roughness. f) Optimal growth recipe for CNTs grown with 99.5% pure ethylene using 99.95% pure ethylene showing minimal sidewall roughness.

Note: Images c, d, e, and f were taken at a 50° tilt.

Although the SEM pictures show the smallest tears and least bowing in sidewalls for the standard recipe for CNT growth with 99.95% pure ethylene, this seems to be a direct result of the shorter growth height of the forest. Furthermore, the sidewall roughness of each run indicates that the optimal growth recipe found for CNT growth with 99.5% pure ethylene produces a higher quality growth when using 99.95% pure ethylene also.

CONCLUSION

The optimal growth recipe, or recipe providing the fastest CNT growth rates while maintaining uniformity of growth throughout CNT structures, for CNT growth using 99.5% pure ethylene uses a 0.304 nm thick iron catalyst, a 190 sccm hydrogen flow-rate, and a 174 sccm ethylene flow-rate at 770 °C. This recipe yields an average growth of 1146 μm in 10 minutes with very uniform growth. This recipe, like all the other tested recipes in the study, grows CNT forests with undesirable features including non-straight growth, bumpy sidewalls, and tears in the structures. Although these undesirable features indicate poor quality growth of CNT forests, the features of this recipe are considerably better than that of the only other recipe which supported fast growth-rates with uniform growth. These poor quality structural features existed not only with the CNT forests grown with 99.5% ethylene purity, but they were also commonplace in CNT forests grown with 99.95% pure ethylene from the same fabricated wafer. This indicates that it is the iron layer thickness, not than the ethylene purity or the gas flow rate, which caused the undesirable features within the CNT forests. If thicker iron catalyst layers are used, the new optimal growth recipe for CNT growth using 99.5% pure ethylene could be used as a reliable option for CNT applications including constructing MEMS devices. It is suggested that a permanent switch to 99.5% pure ethylene would be most effective in creating growth sufficient in height and quality for future applications because it yields a faster growth rate while maintaining similar growth features.

The data suggests that the growth parameters for hydrogen and ethylene flow-rates are not linked due to the faster and more uniform growth rates experienced when hydrogen flow-rates are at 190 sccm regardless of ethylene flow-rates. Growth-rate and uniformity of CNT forests increased as ethylene flow-rates increased independent of hydrogen flow-rate and iron thickness. The growth-rate increased as the ethylene flow-rate was increased up to 174 sccm; however, it is uncertain how growth would be affected with ethylene flow-rates above 174 sccm. The iron catalyst thickness also seems to be independent of the other factors as is evident through the effects iron thickness has on the CNT forest features. This is most easily seen by the existence of craters on the surface of all runs with an iron catalyst thickness of 0.507 nm which are not present on any other runs.

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