Determining the Ideal Growth Kinetics of Carbon Nanotubes

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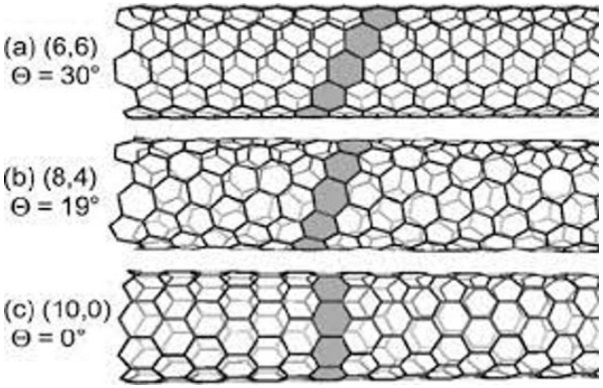
Background

- Carbon nanotubes (CNTs) can carry 550,000 times more current per area than copper
- CNTs are extremely efficient conductors of electricity but are difficult to consistently produce in uniform samples with controlled diameter
- Historically made with iron, which is unstable, so higher melting point element that is stable is necessary
- Research project aimed to study optimal growth conditions
- Bimetal alloy nanoparticle consisting of high melting metal and an iron group metal in 4 sample sizes XS, S, M, and XL, whose diameter were 1.2-3 nm, were spin-coated on a silicon wafer and annealed with H_2 for 0, 10, 20, and 30 minutes and then studied for changes
- Particle evolution on the samples was then studied with an Atomic Force Microscope (AFM) and their nanotube density and diameters were recorded
- AFM uses a laser reflected off a cantilever to determine the height of the catalyst particles

Atomic Force Microscope



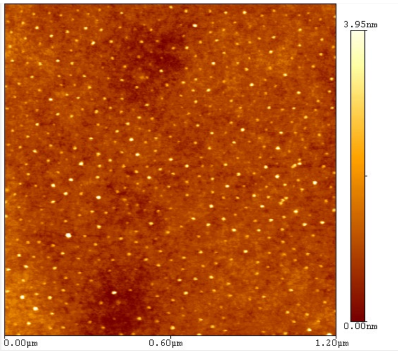
Semiconducting CNTs

Sample Name: CAT 1215202201 XS
Reduction Temperature: 900ºC:

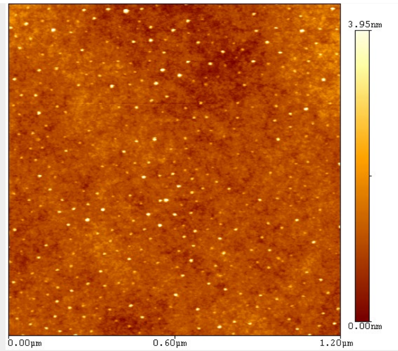
Time	0 min	10 min	20 min	30 min

AFM Raw Image

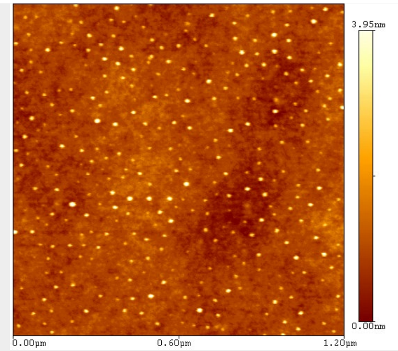
0 min



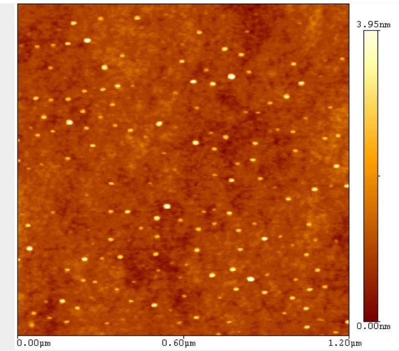
10 min



20 min

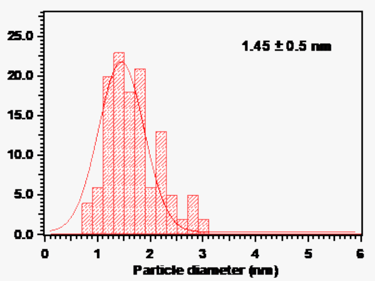


30 min

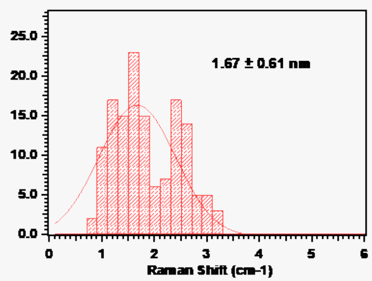


Synthesis

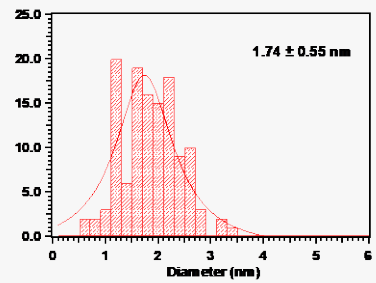
0 min



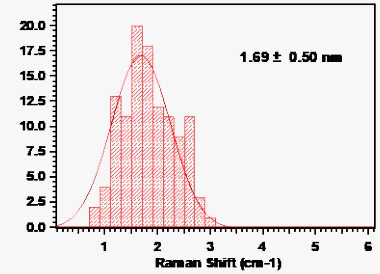
10 min



20 min



30 min

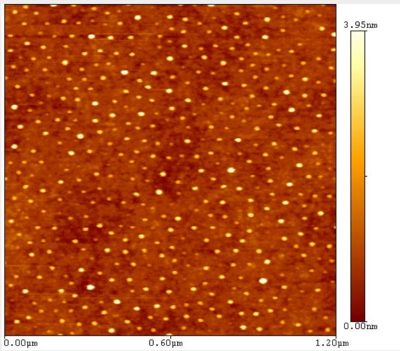
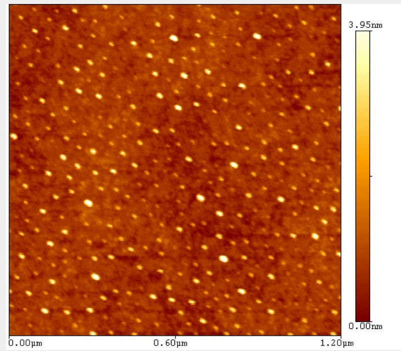
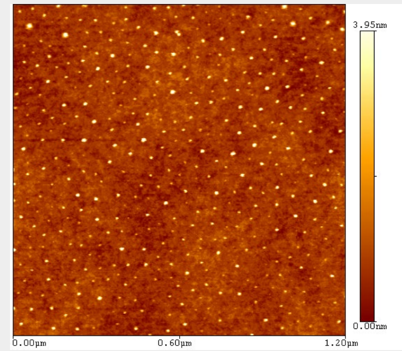
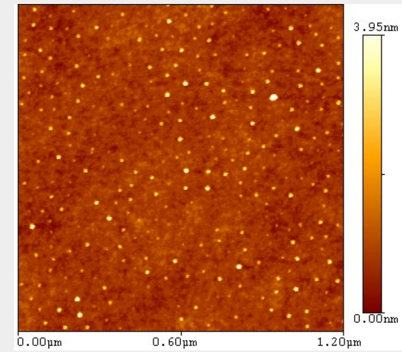
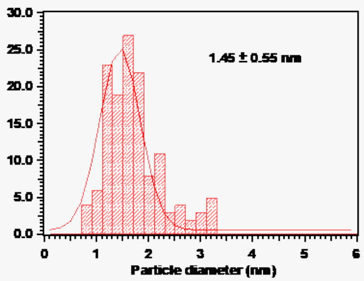
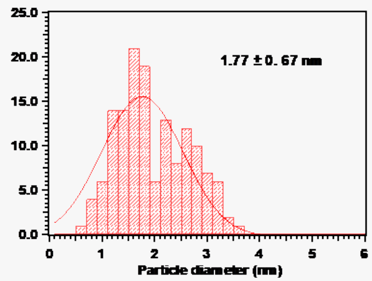
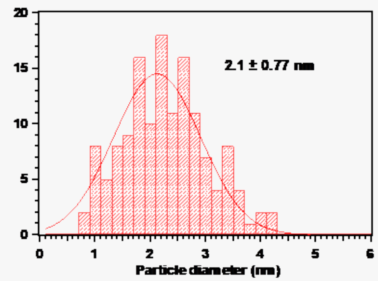
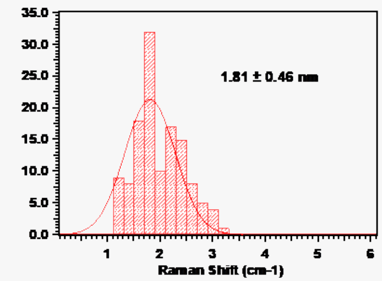


	0 min	10 min	20 min	30 min
Particle Density in um				
	~337 / um^2	~282 / um^2	~231 / um^2	~142 / um^2
Fitted Gaussian Peak Nanotube Diameter				
	1.45	1.67	1.74	1.69
Standard deviation in nm				
	+/- 0.50 nm	+/- 0.61 nm	+/- 0.55 nm	+/- 0.50 nm

Peak

Not an ideal standard deviation
Lacks uniformity

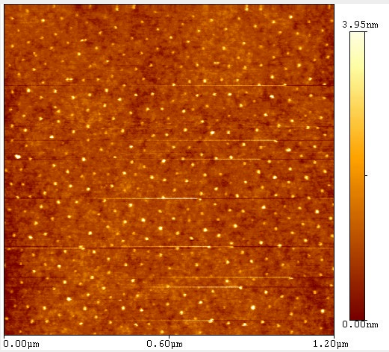
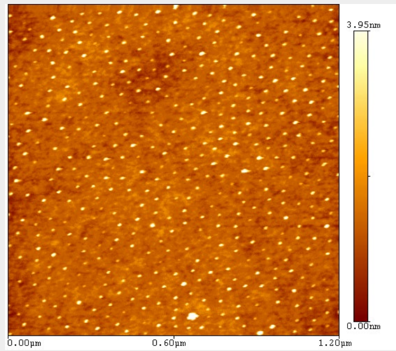
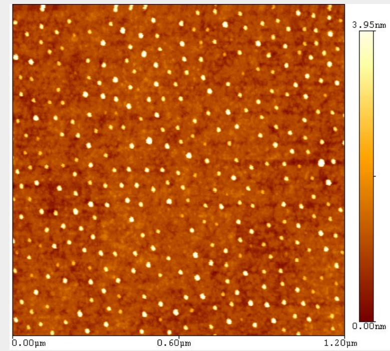
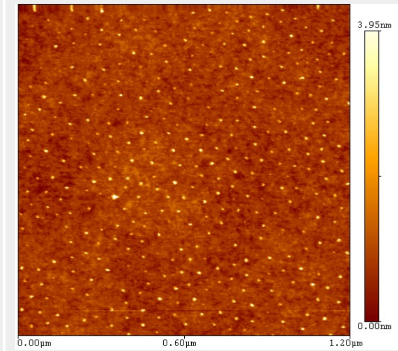
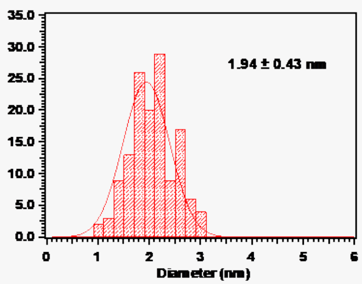
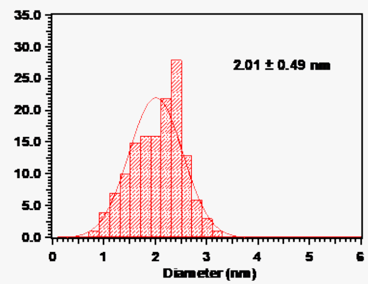
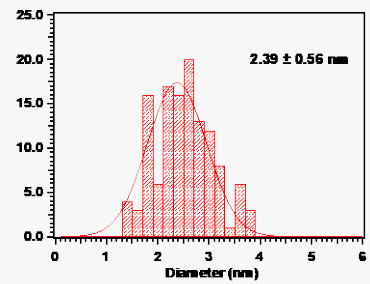
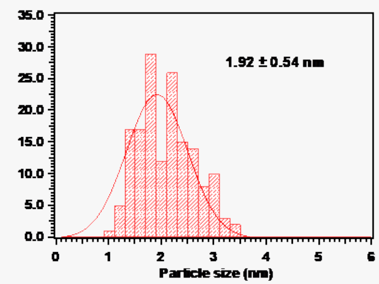
Sample Name: CAT 0208202302 S
Reduction Temperature: 900ºC:

Time	0 min	10 min	20 min	30 min
AFM Raw Image				
Synthesis				
	Particle Density in um			
	~291 / um^2	~247 / um^2	~302 / um^2	~206 / um^2
	Fitted Gaussian Peak Nanotube Diameter			
	1.45	1.77	2.10	1.81
	Standard deviation in nm			
	+/- 0.55 nm	+/- 0.67 nm	+/- 0.77 nm	+/- 0.46 nm

Peak

Relatively high standard deviation indicates lack of uniformity

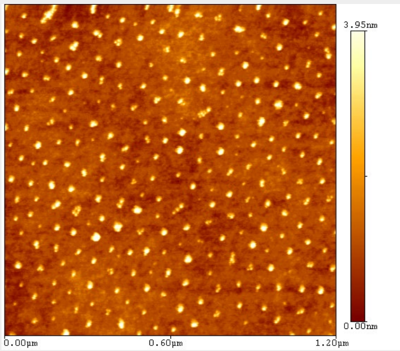
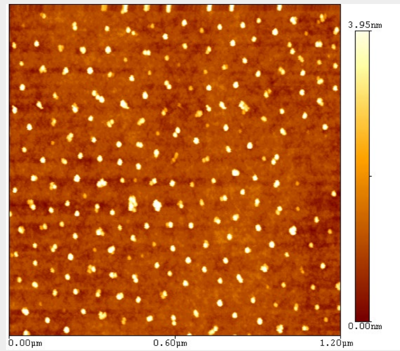
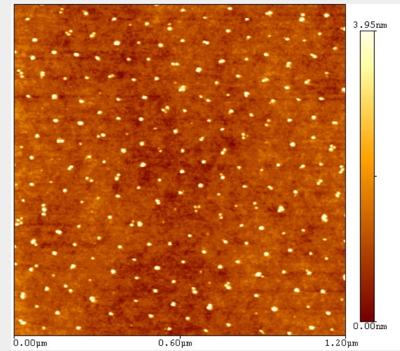
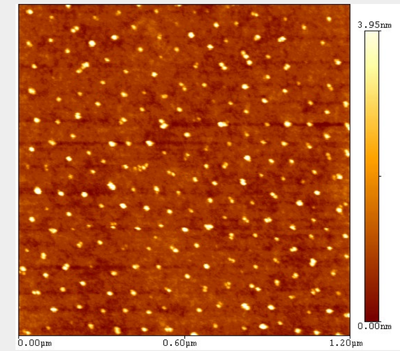
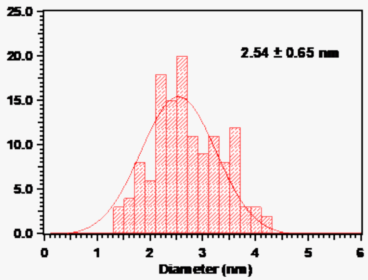
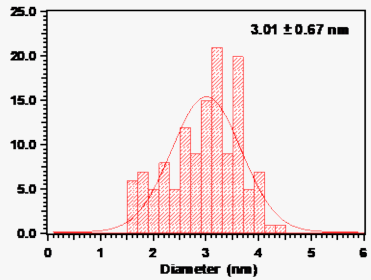
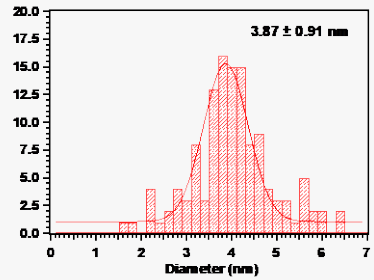
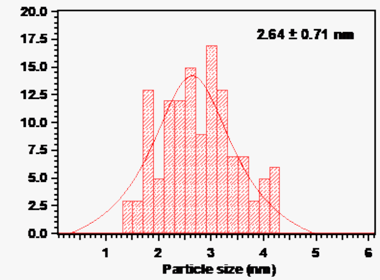
Sample Name: CAT 1025202203 M1
Reduction Temperature: 900ºC:

Time	0 min	10 min	20 min	30 min
AFM Raw Image				
Synthesis				
	Particle Density in um			
	~300 / um^2	~323 / um^2	~235 / um^2	~252 / um^2
	Fitted Gaussian Peak Nanotube Diameter			
	1.94	2.01	2.39	1.92
	Standard deviation in nm			
	+/- 0.43 nm	+/- 0.49 nm	+/- 0.56 nm	+/- 0.54 nm

Peak

Relatively low standard deviation indicates uniformity and stability

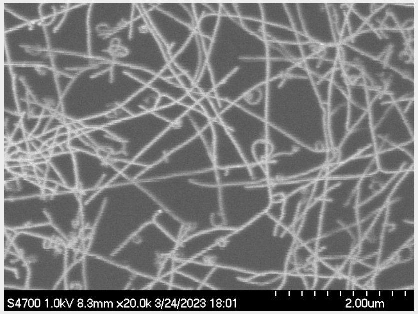
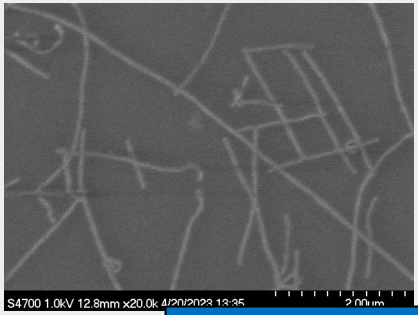
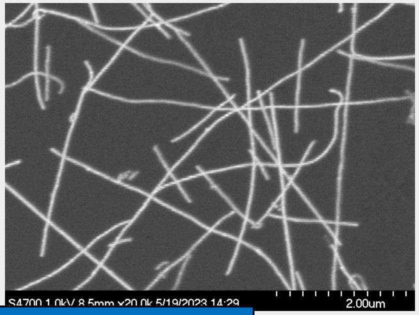
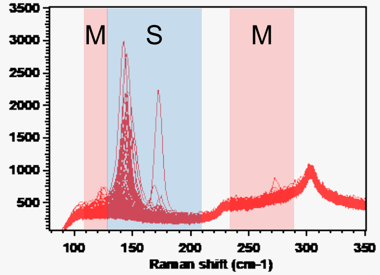
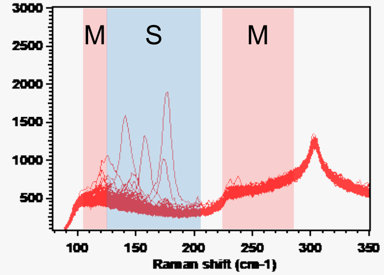
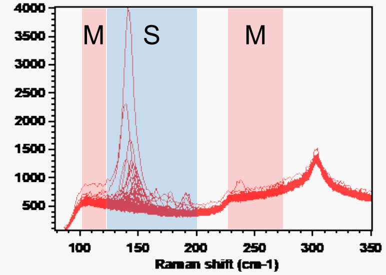
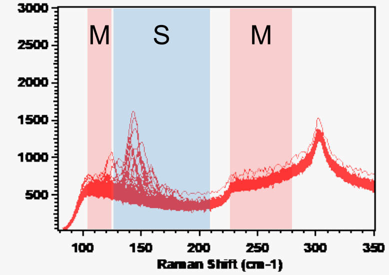
Sample Name: CAT 1215202203 XL
Reduction Temperature: 900ºC:

Time	0 min	10 min	20 min	30 min
AFM Raw Image				
Synthesis				
	Particle Density in um			
	~233 / um^2	~208 / um^2	~213 / um^2	~207 / um^2
	Fitted Gaussian Peak Nanotube Diameter			
	2.54	3.01	3.87	2.64
	Standard deviation in nm			
	+/- 0.65 nm	+/- 0.67 nm	+/- 0.91 nm	+/- 0.71 nm

Peak

Relatively low standard deviation indicates uniformity

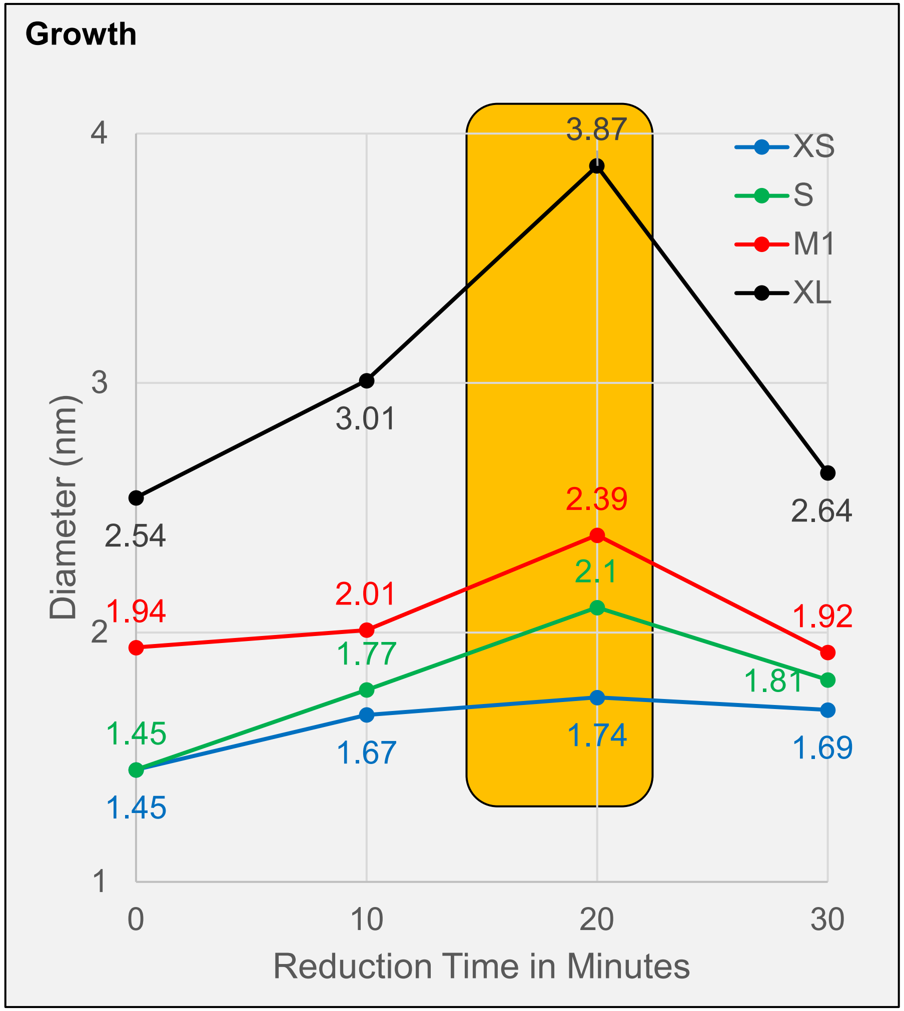
Chemical Vapor Deposition (CVD) Results for the Growth of Nanotubes for the M1 Silicon Wafer Catalyst

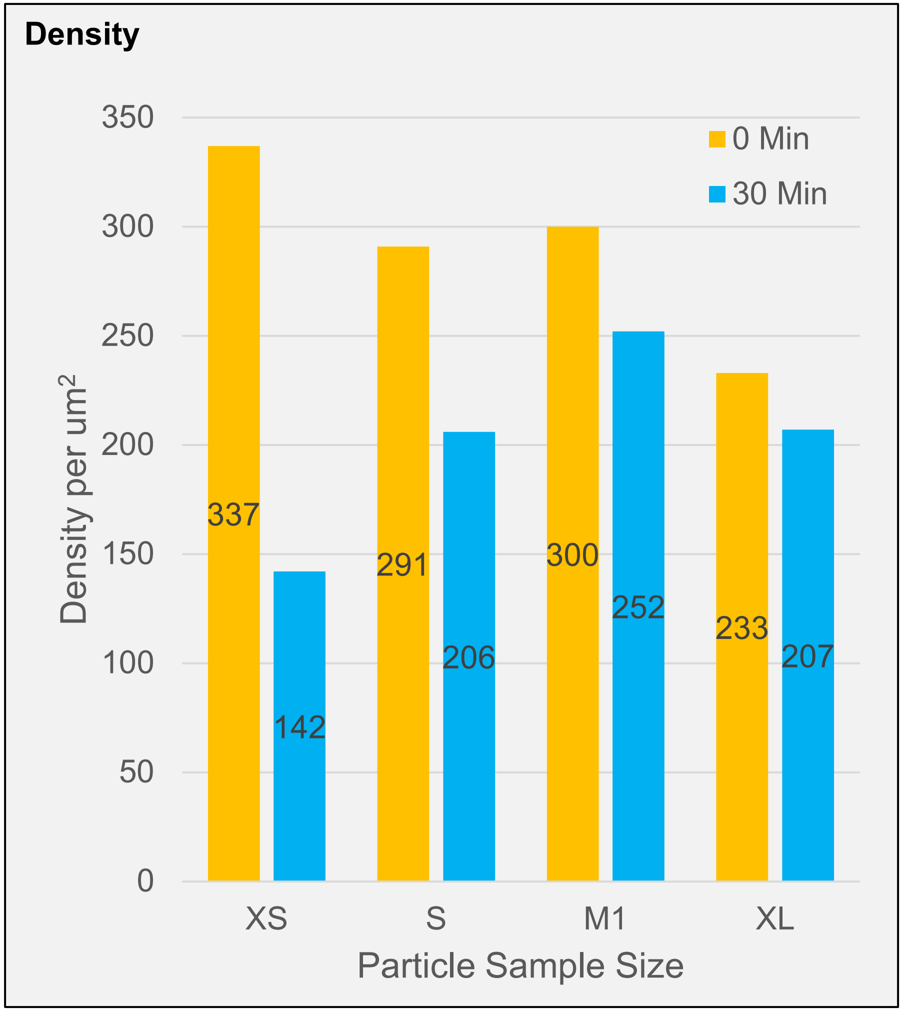
	Sample: # 03.23.2023 - 01	Sample: # 04.14.2023 - 01	Sample: # 04.17.2023 - 01	Sample: # 05.17.2023 - 01
Raw image				
Synthesis				
	Scanning Electron Microscope Density Data			
	~10 / um^2	~8 / um^2	~5 / um^2	~4 / um^2
	H_2 reduction over time at a temperature of 900ºC			
	0 min	10 min	20 min	30 min

Raman shows diameter control and selectivity for M1

Note: The formula for calculating the diameter of a nanotube is d_{cnt}= 248 / raman shift

Catalyst Particle Diameter Growth & Density Evolution





- Regardless of sample size, all batches peaked at around 20 minutes
- M1 diameter is the most stable from 0 to 30 minutes

- In all 4 sample sizes, the particle density decreased over time
- Smallest drops: M1 had a 16% drop and XL had a 12% drop

Conclusion & Next Steps

1. The data suggests that the diameter of the nanotubes peaks at about 20 minutes of reduction time at a temperature of 900ºC
2. The particle density steadily decreases as reduction time increases in all samples
3. Density remains the least unchanged in M1 and XL indicating stability, but M1 has the most stable diameter
4. Many samples had a relatively larger standard deviation making statistical significance difficult to determine
5. Catalyst is evolving, but exact causes are not fully understood

Next steps:

1. Data for 40 minute reduction time can be collected
2. Try a higher melting point metal and investigate its thermal stability
3. Image the substrate with a Transmission Electron Microscope