Using TGA for Quick Evaluation of Few-Walled Carbon Nanotubes

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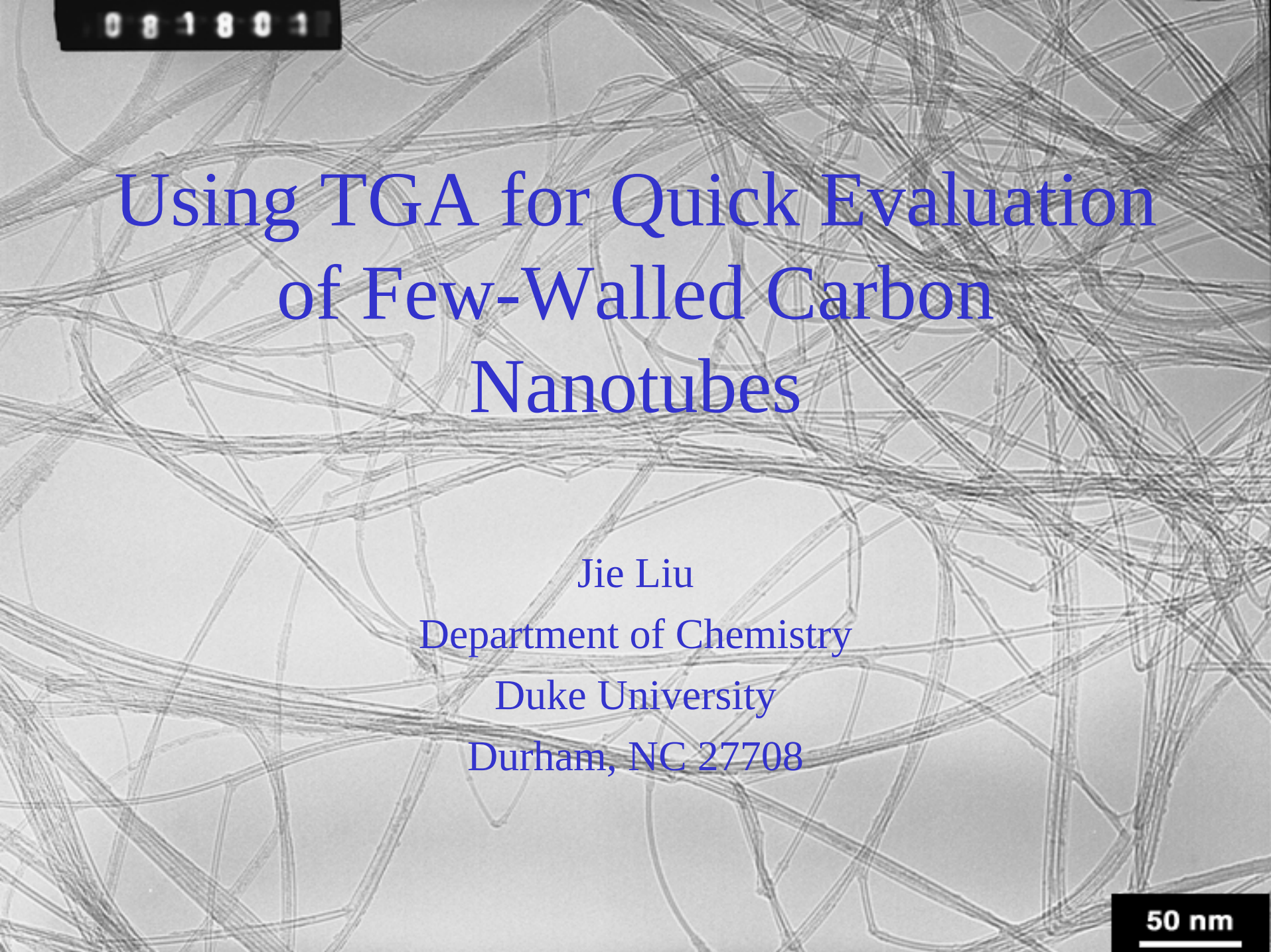
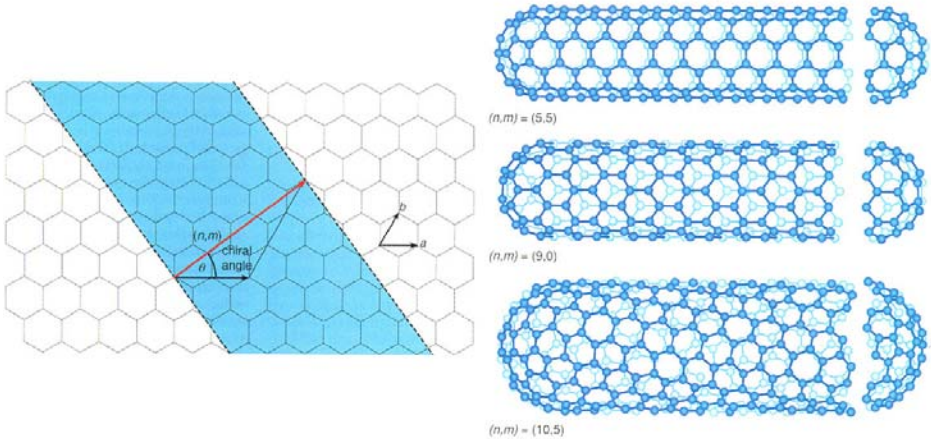
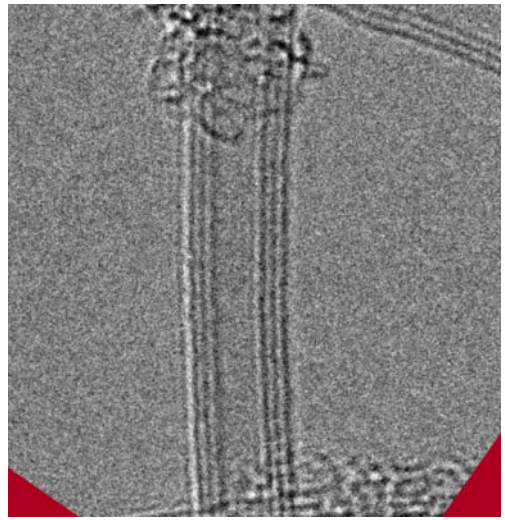


Introducing Carbon Nanotube





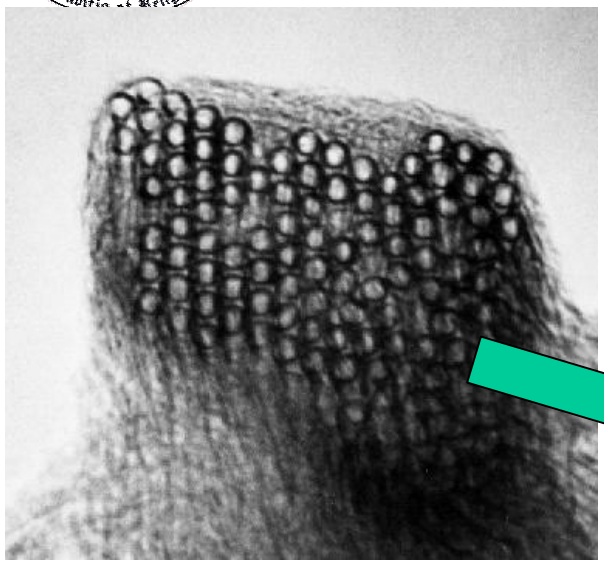
Unique Properties of CNTs:

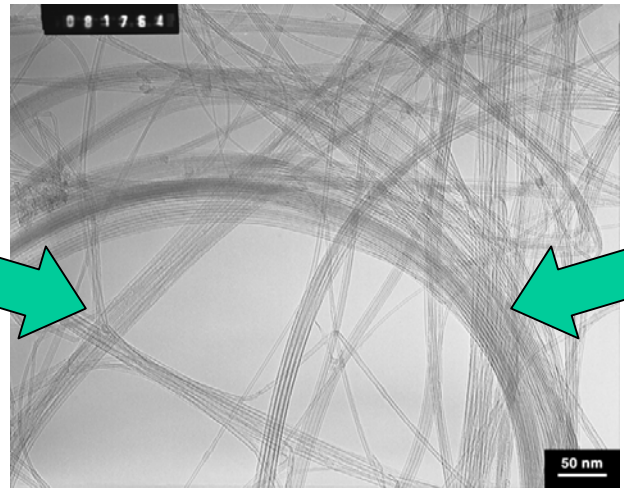
1. Electronic properties: Can be either metallic of semiconducting.
2. Mechanical Properties: Strongest nanoscale fiber.
3. High surface area.
4. Good chemical stability.
5. Low working function.

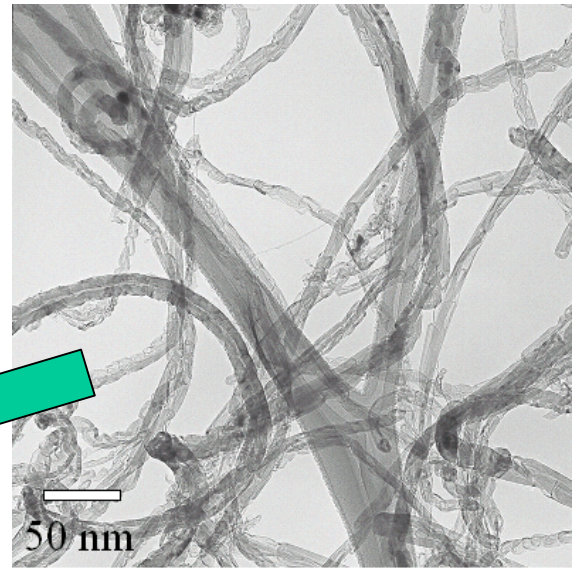
Major Applications

1. High Strength Composite Materials:
2. Nanoscale Electronics and Sensors.
3. Field Emitters: Flat panel display, x-ray, TV etc.
4. Electrode Material: batteries, capacitors,fuel cells etc.

What are FWNTs?







SWNTs

1. One layer of carbon
2. Low defect density
3. Good mechanical and electrical properties
4. Hard to prepare
5. Hard to purify

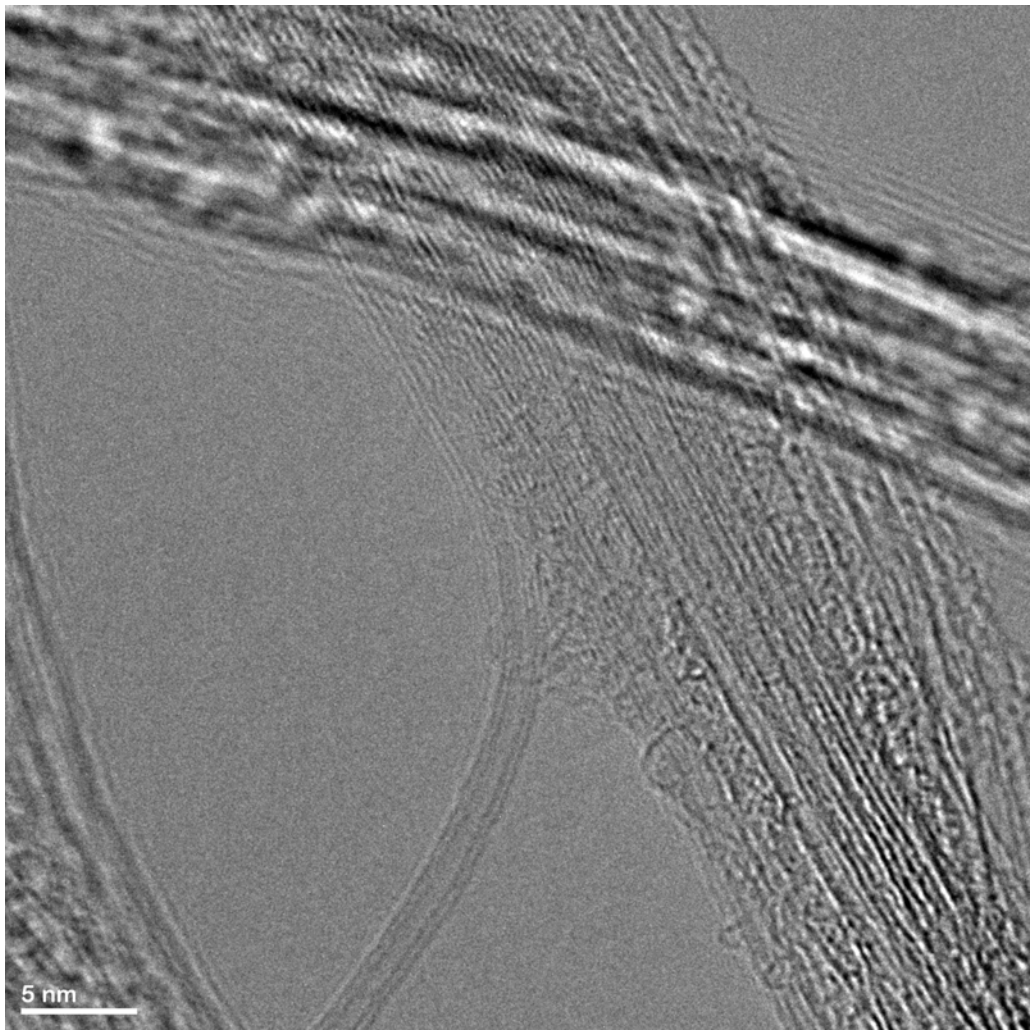
FWNTs

1. 2-5 layers of carbon
2. Low defect density
3. Similar to SWNTs in mechanical properties
4. Unclear electric properties
5. Easy to prepare
6. Easy to purify

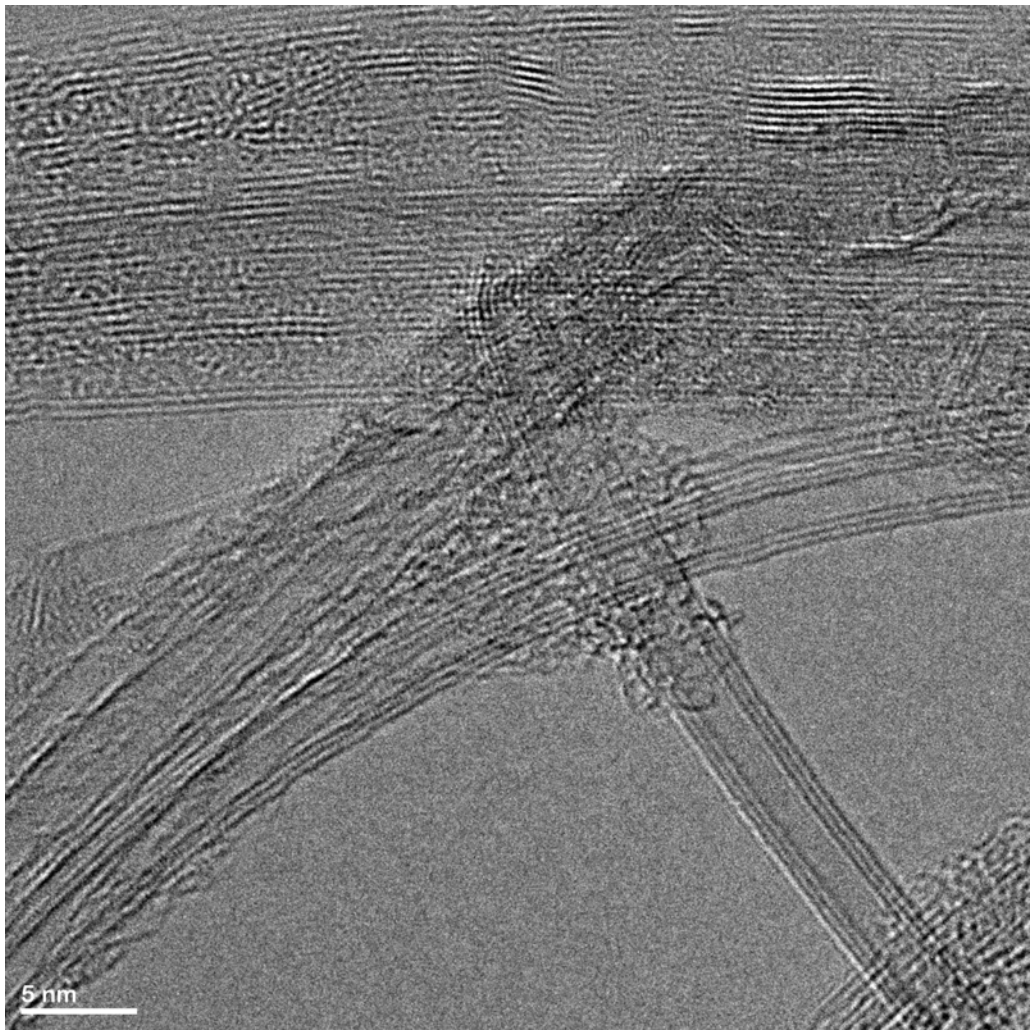
MWNTs

1. Many layers of carbon
2. Normally High defect density
3. Not as good as SWNTs in mechanical and electrical properties
4. Easy to Prepare
5. Easy to Purify.

Doubled Walled CNT



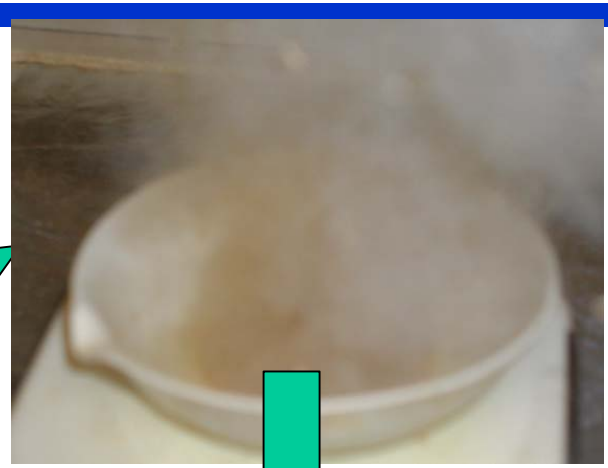
Triple-Walled CNTs



Large Scale Synthesis

Metal Nitrate (Mg, Fe, Co, Mo etc.)
Glycine, Water

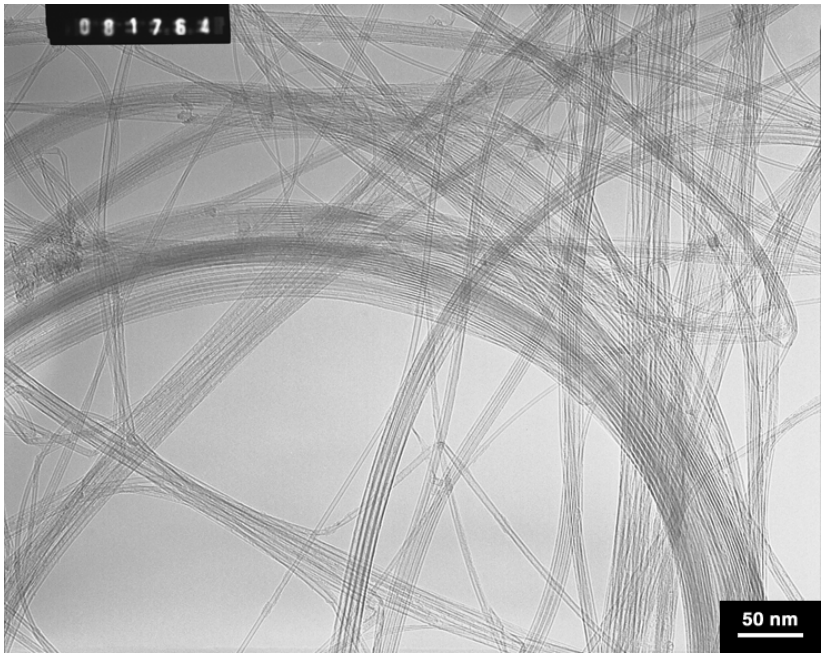
Heating in Air

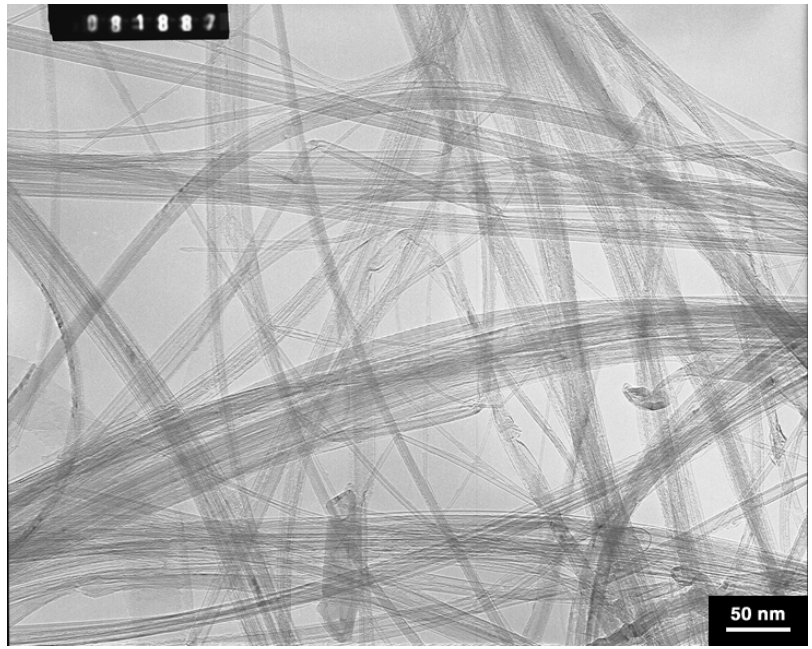


Catalyst

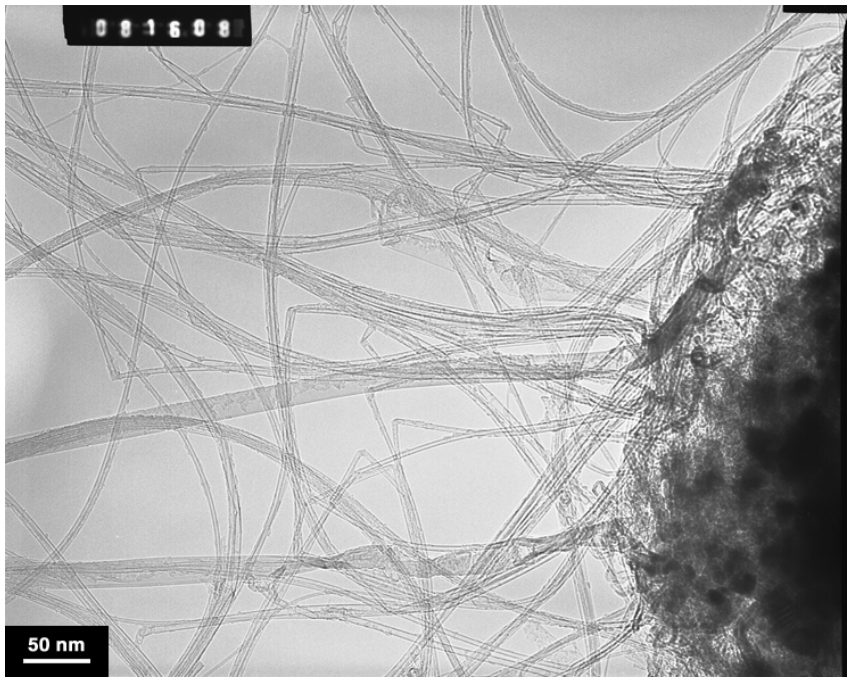
Surface area ~ 110 m^2/g

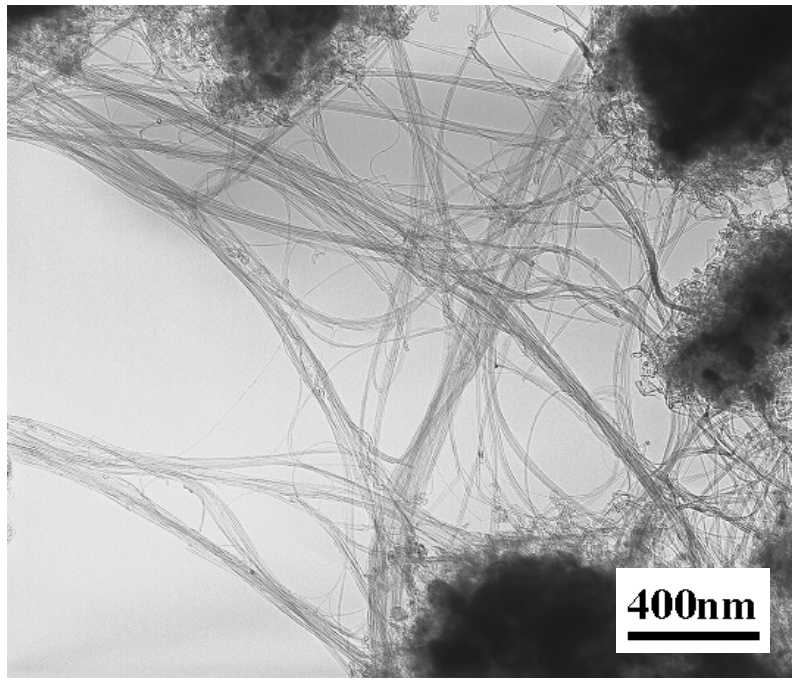
High Quality Samples





Normal Samples



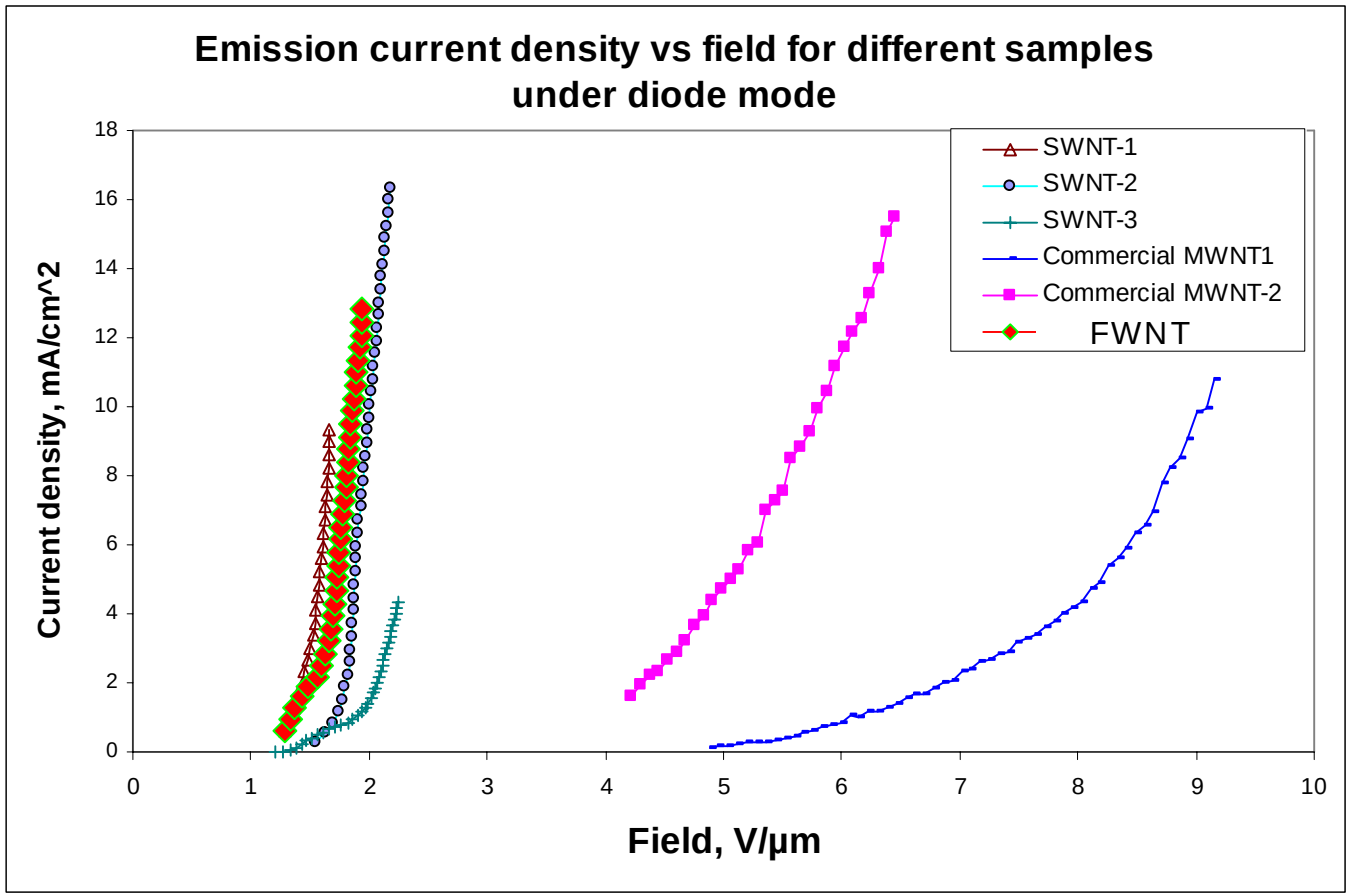


Comparison with SWNTs

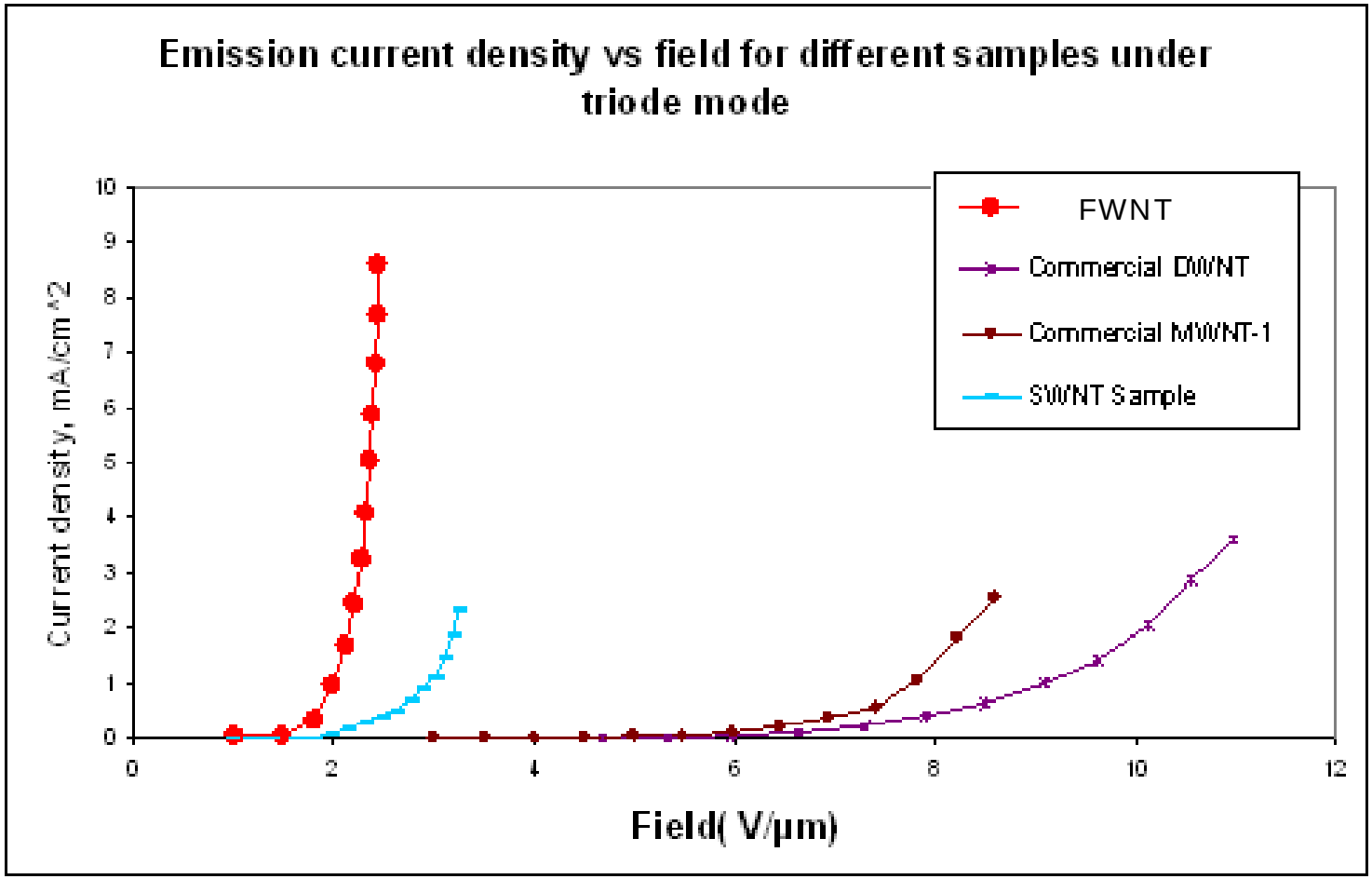
FWNTs	SWNTs
2-5 layer of side walls	Single layer of side walls
Diameter range from 2 to 10 nm. Average diameter ~5 nm	Diameter range between 1 and 3 nm.
Better thermal and chemical stability	
More rigid and do not form large bundles	Tend to form large bundles

Field Emission Properties

In collaboration with Xintek Inc. (Formerly known as ANI)

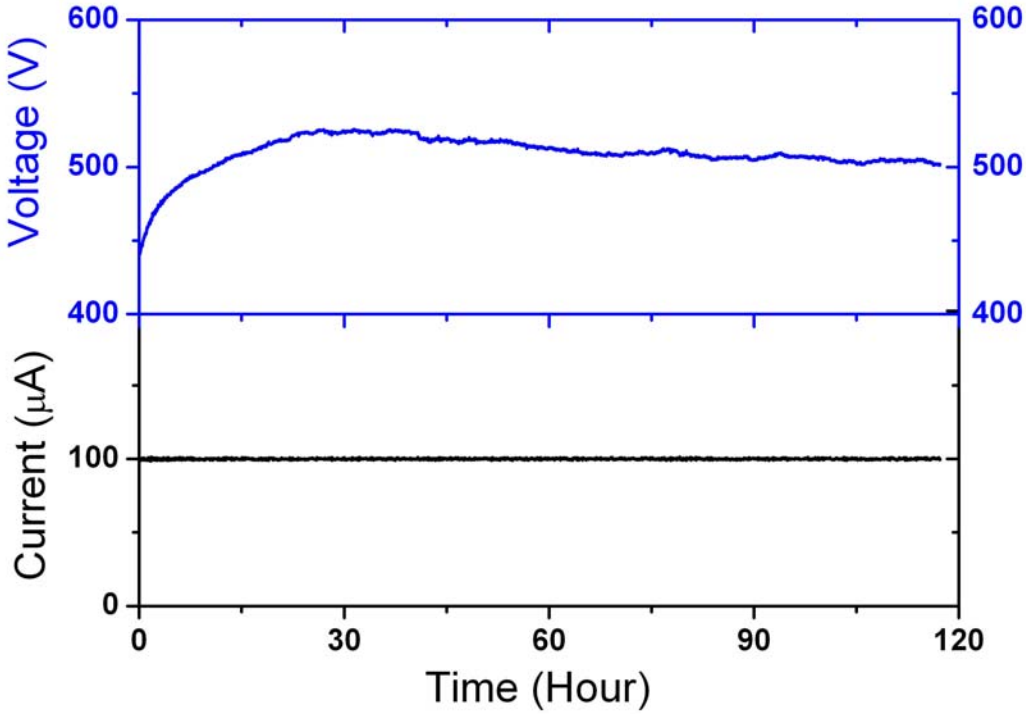


Field Emission Properties



Field Emission Properties

In collaboration with Xintek Inc.

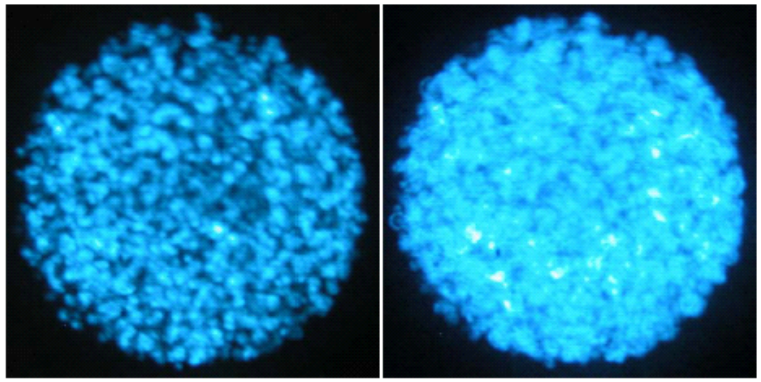


Good long term stability

Field Emission Properties

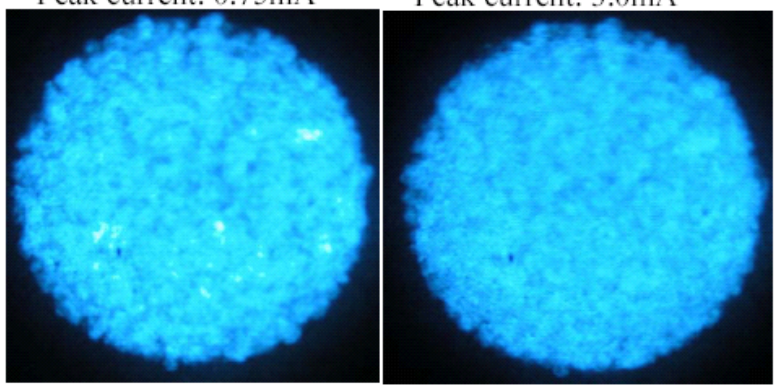
In collaboration with Xintek Inc.

100Hz, 20us
(0.2%)



Voltage: 1500V (1425V)
Peak current: 0.75mA

Voltage: 2000V (1700V)
Peak current: 3.0mA

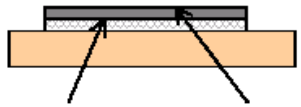


Voltage: 2500V (1900V)
Peak current: 6.0mA

Voltage: 3000V (2100V)
Peak current: 9.0mA

047201-1
1cm in diameter sample

Two layers



Good emission uniformity

Field Emission Properties

In collaboration with Xintek Inc.

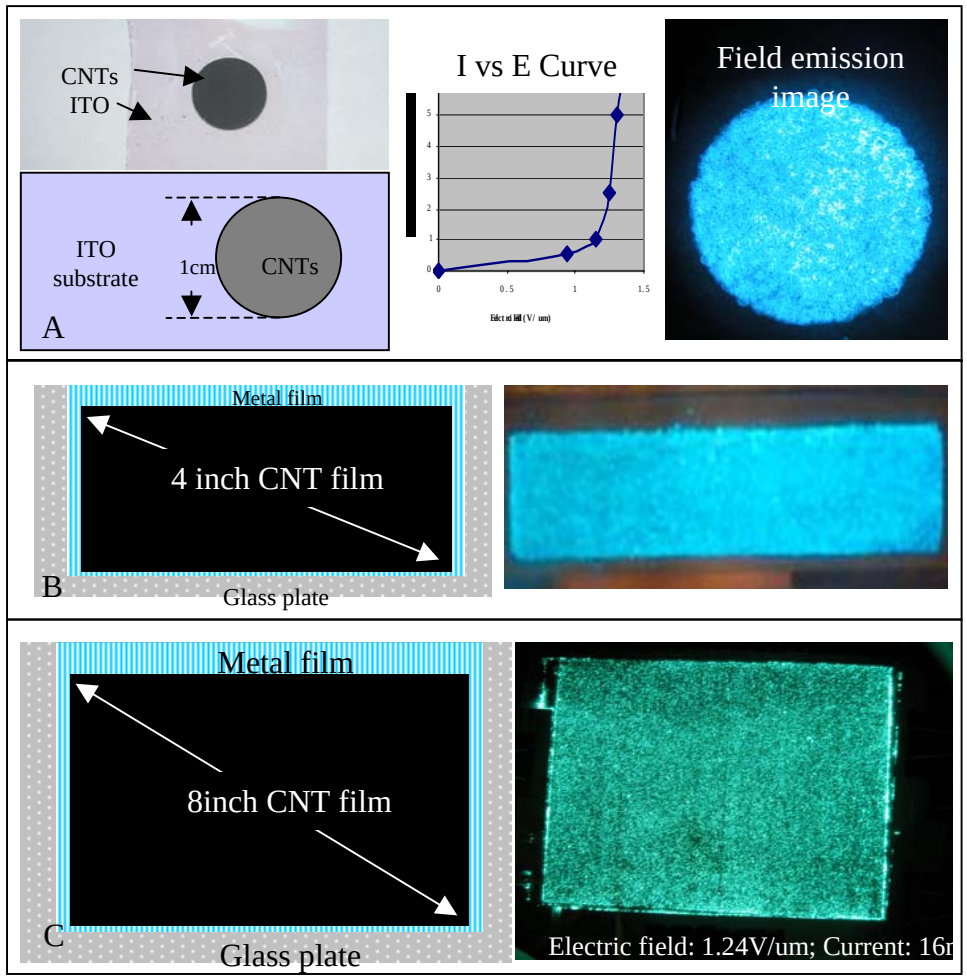
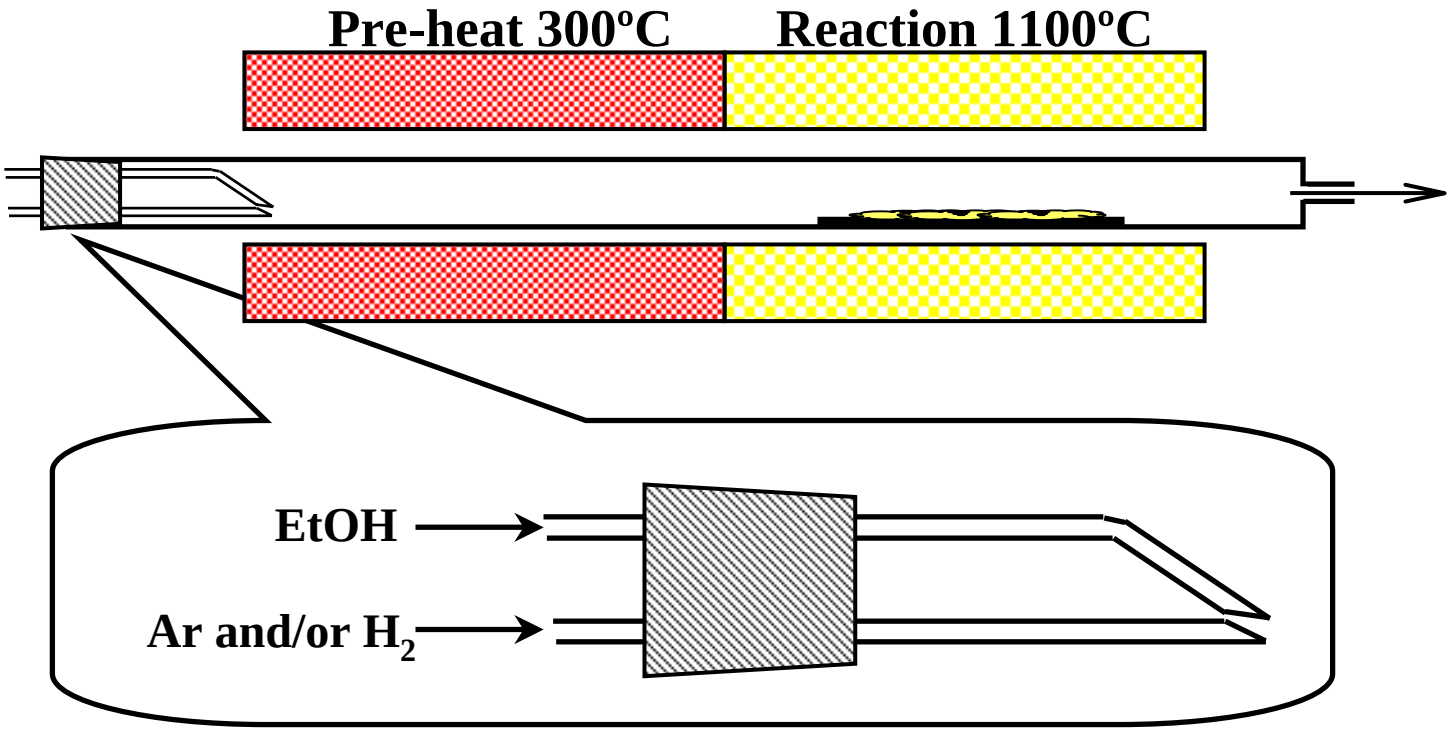


Fig. 10. (A) An optical image, a schematic drawing, an emission current (I) versus electric field (E) curve and a field emission image of a Xintek's 1cm in diameter CNT film on ITO glass substrate. (B) and (C) Schematic drawings and a field emission images of a Xintek's 4'' and 8'' CNT cathode. All the images were taken in pulse mode at 10% duty cycle.

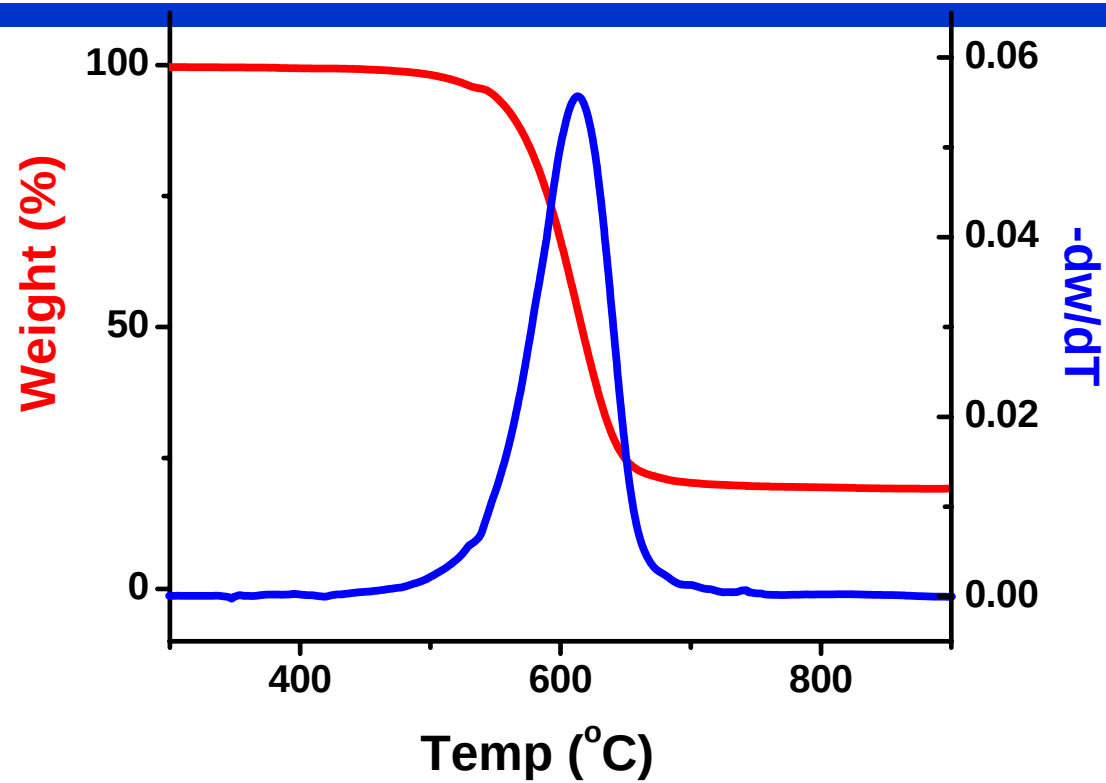
Optimization of Growth Conditions

Experimental Setup



1) Heat under from room temperature to 1100 ºC
2) Turn on EtOH and react for 20 min
3) Turn off EtOH and cool the system down to room temperature

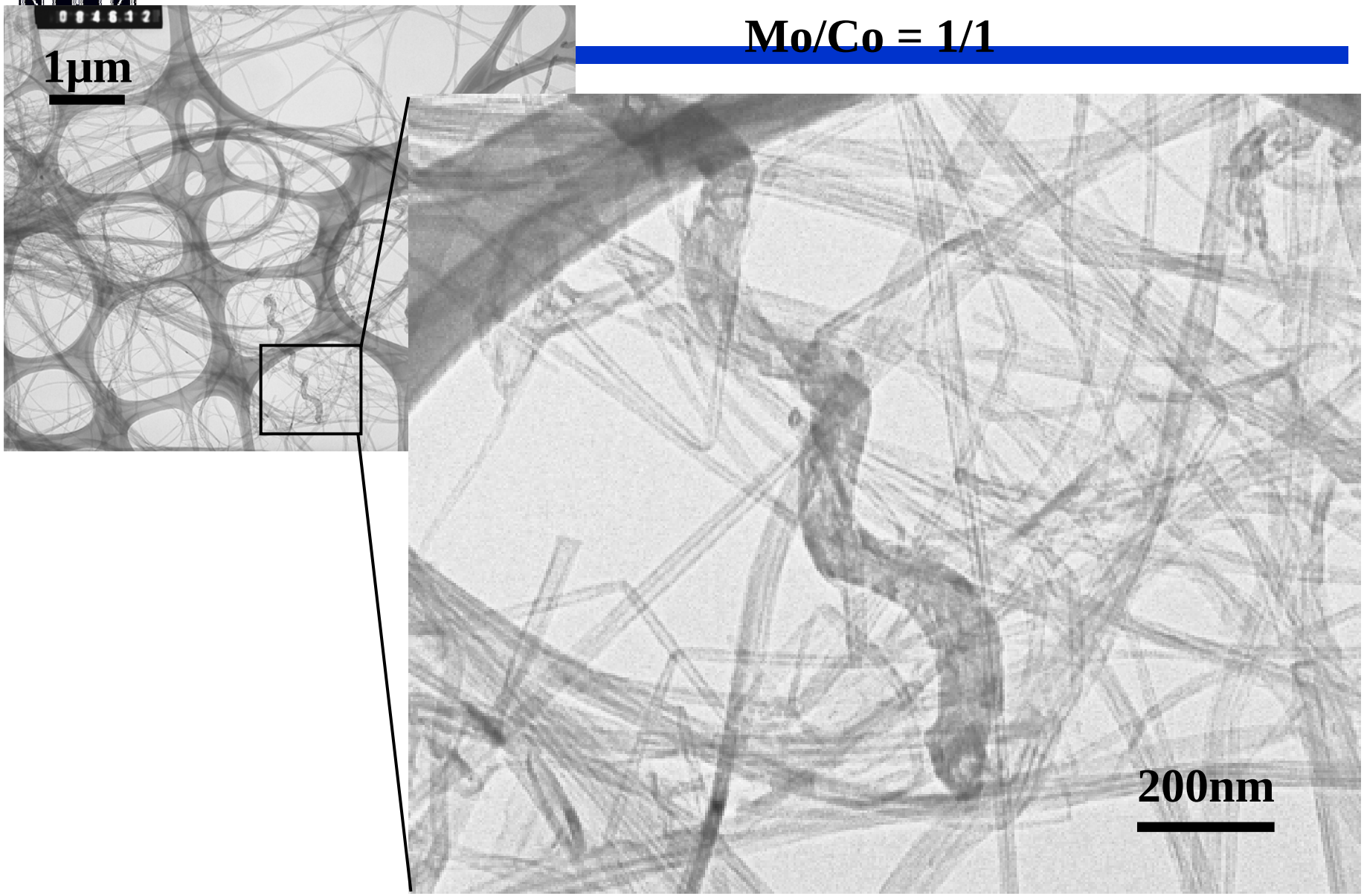
Analysis of TGA Data (



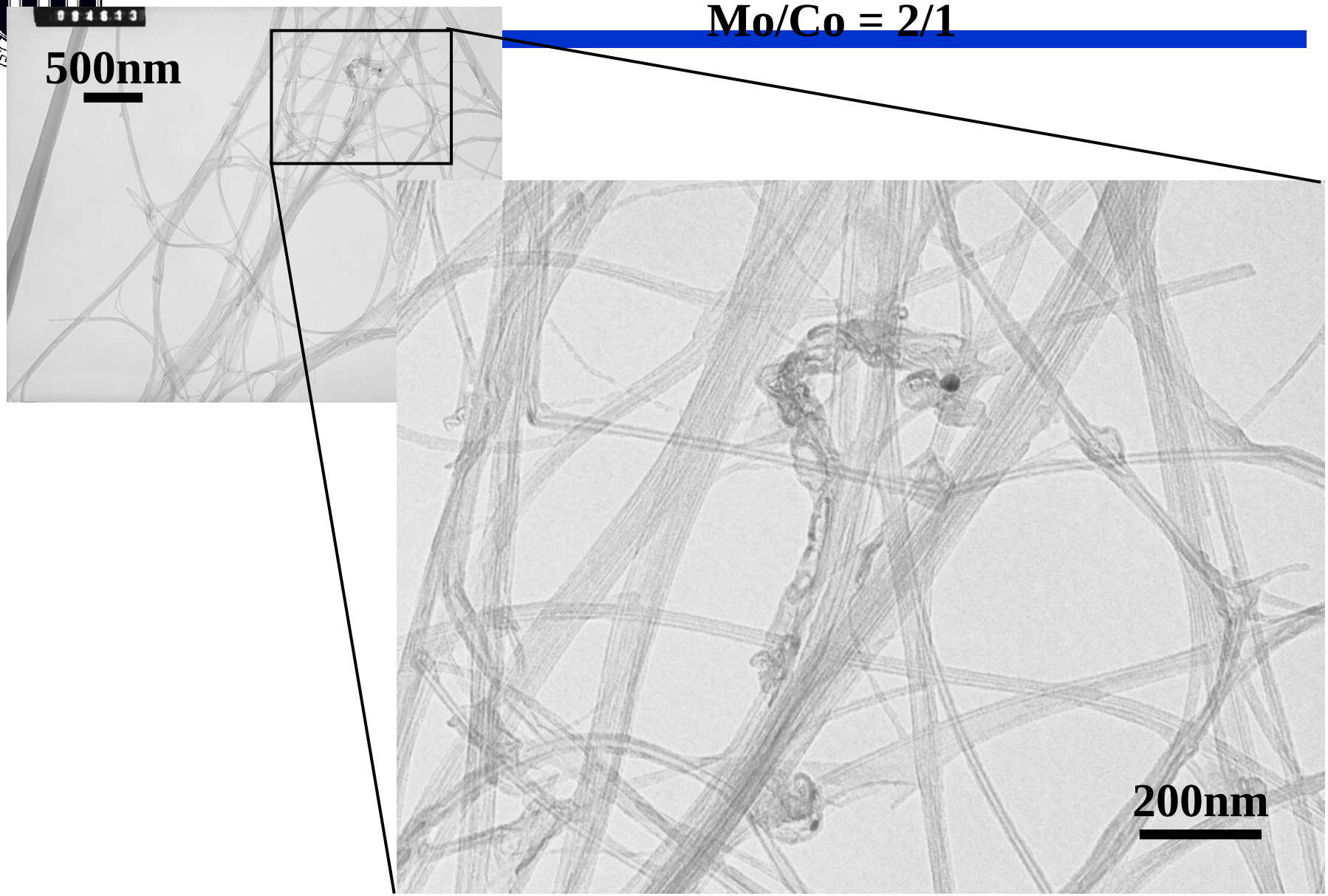
Weight loss plot vs temperature (TGA) **This plot suggests carbon yield of the sample**

First order differentiation of weight loss plot (DTGA) **The peak position of this plot suggests the composition of sample.**

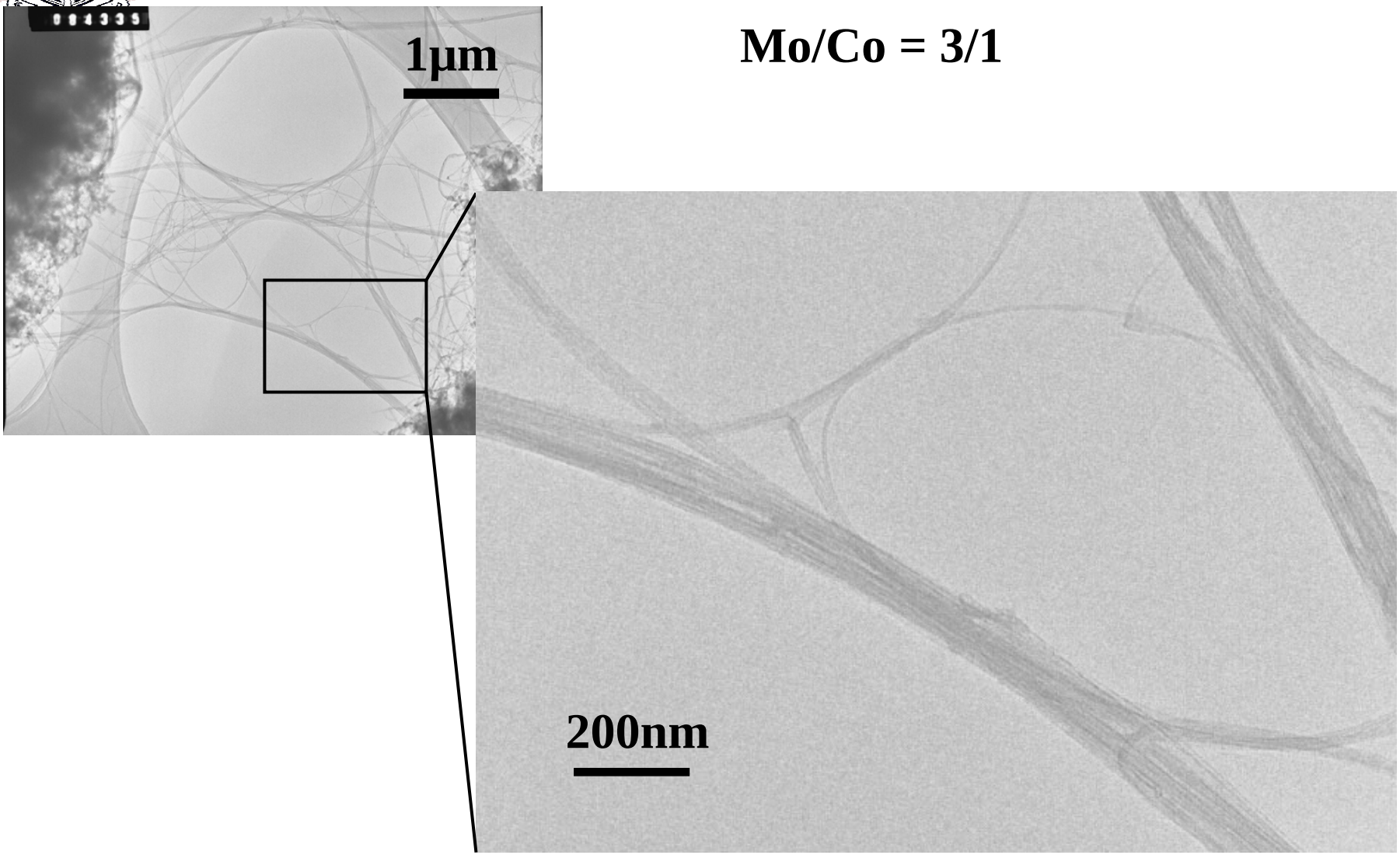
Mo/Co = 1/1

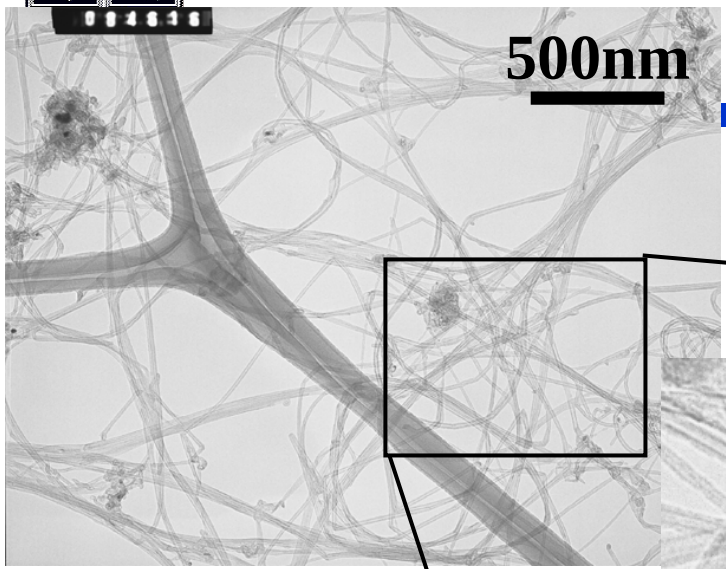


Mo/Co = 2/1

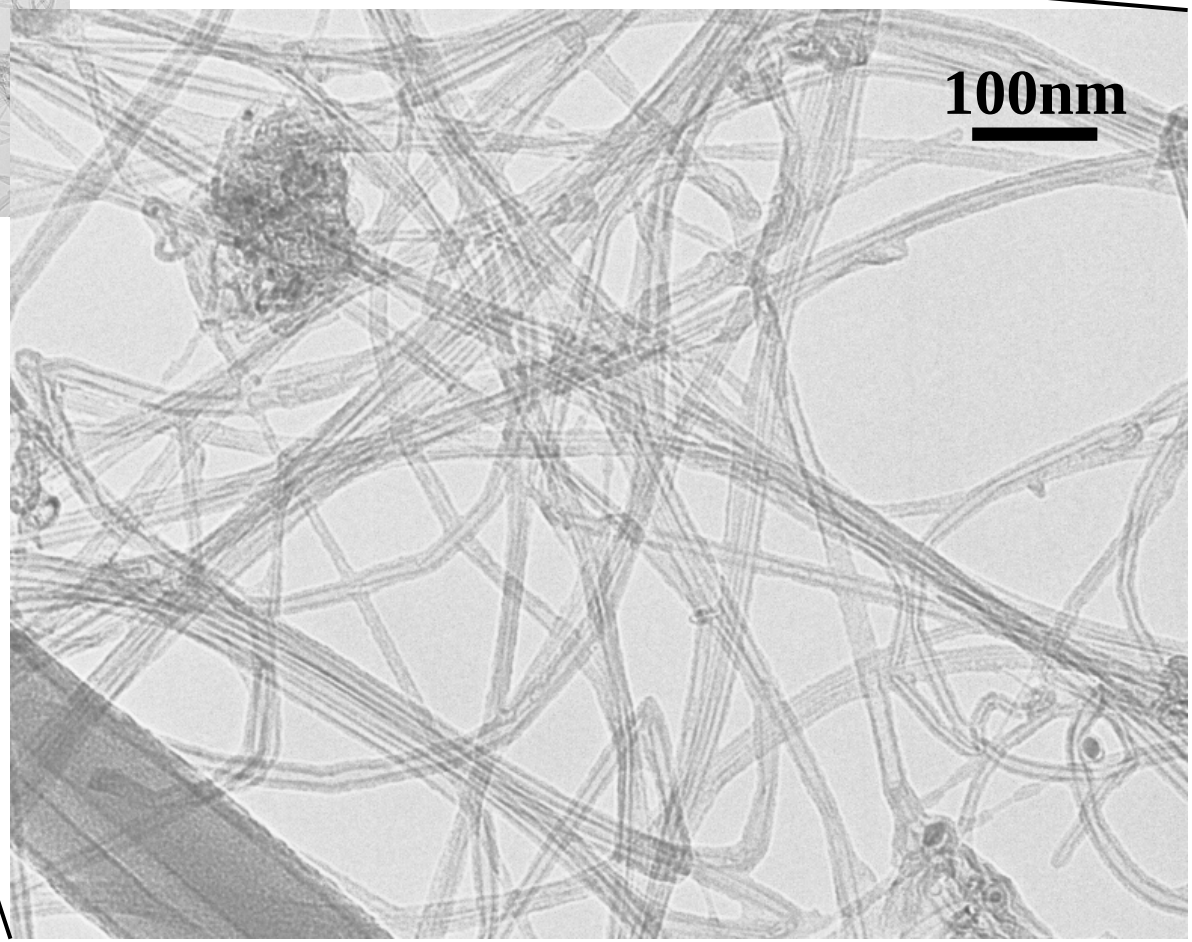


Mo/Co = 3/1

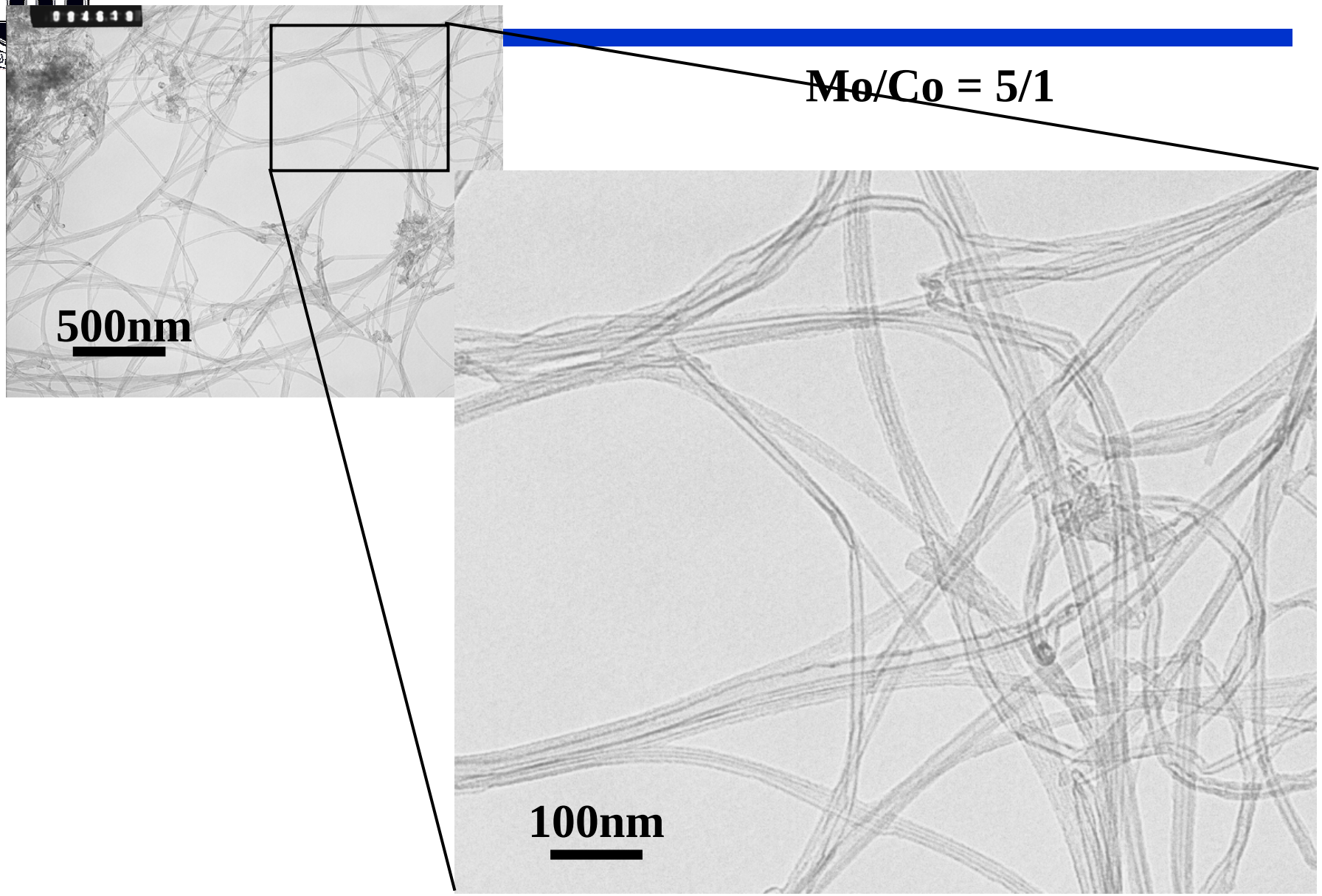




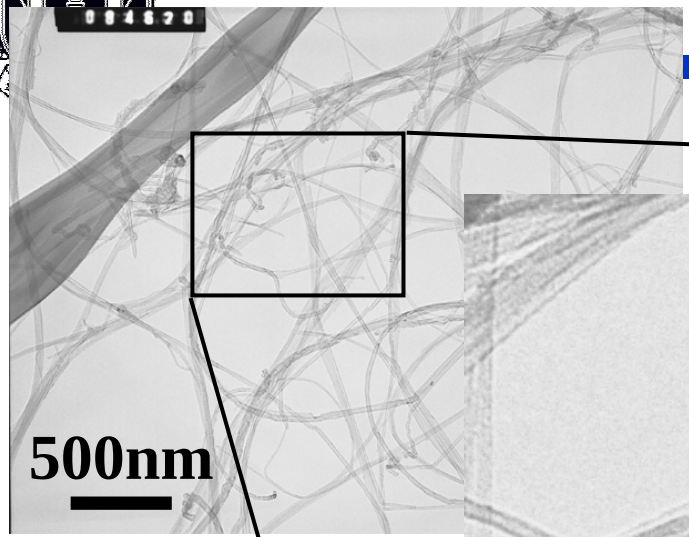
Mo/Co = 4/1

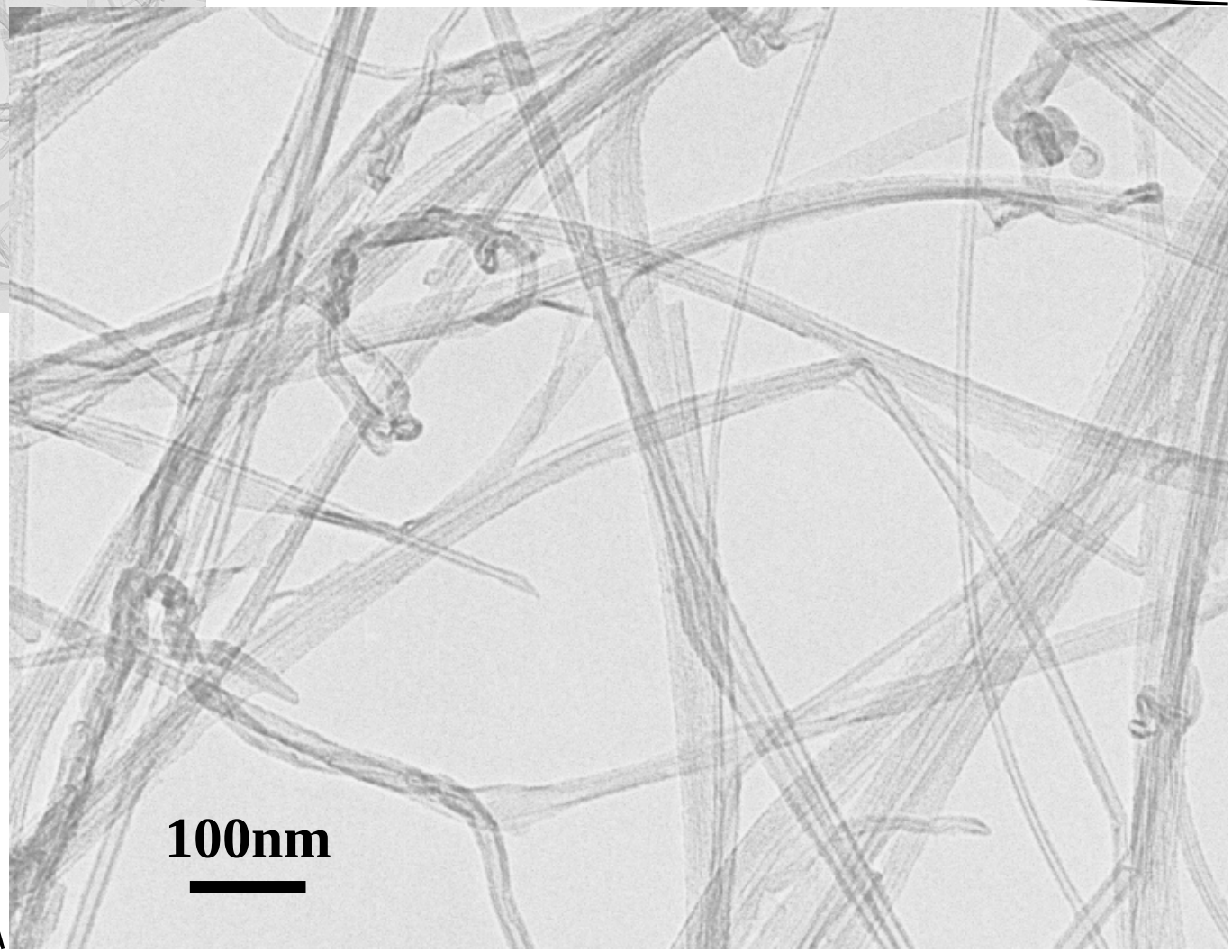


Mo/Co = 5/1

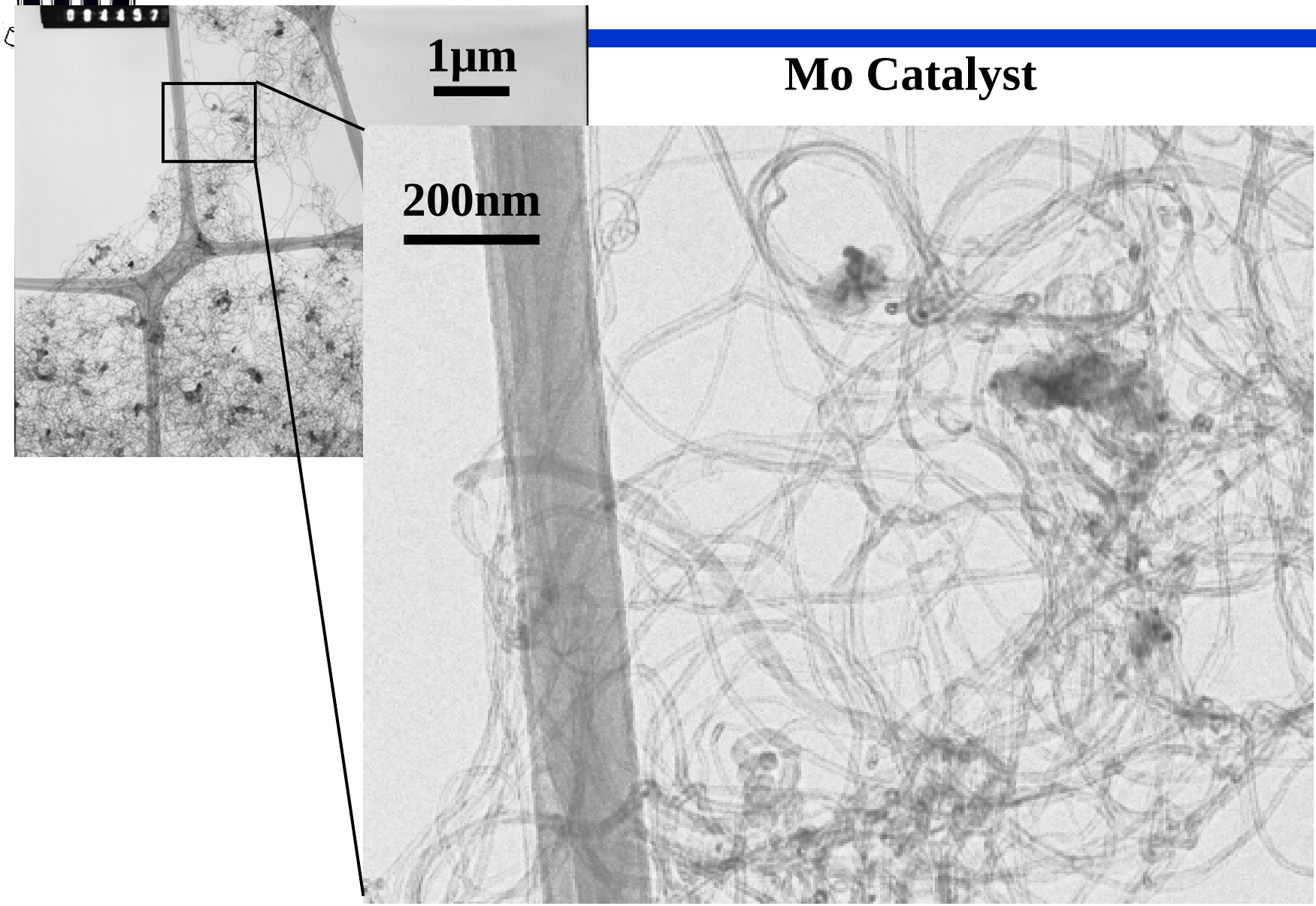


Mo/Co = 6/1



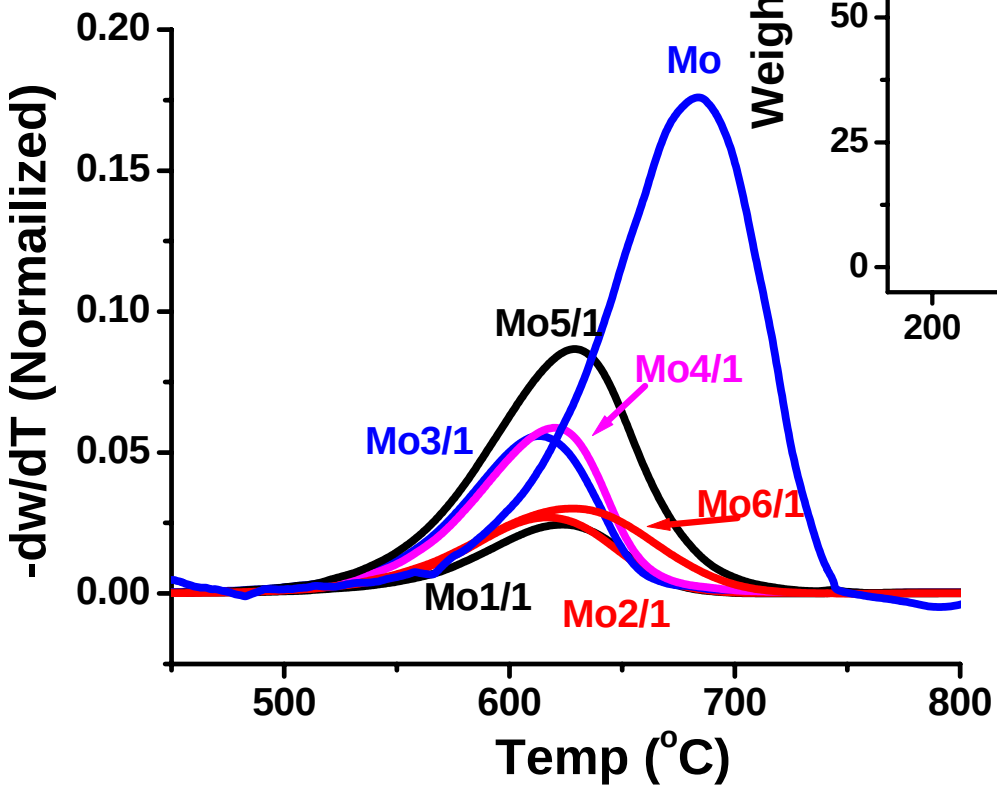


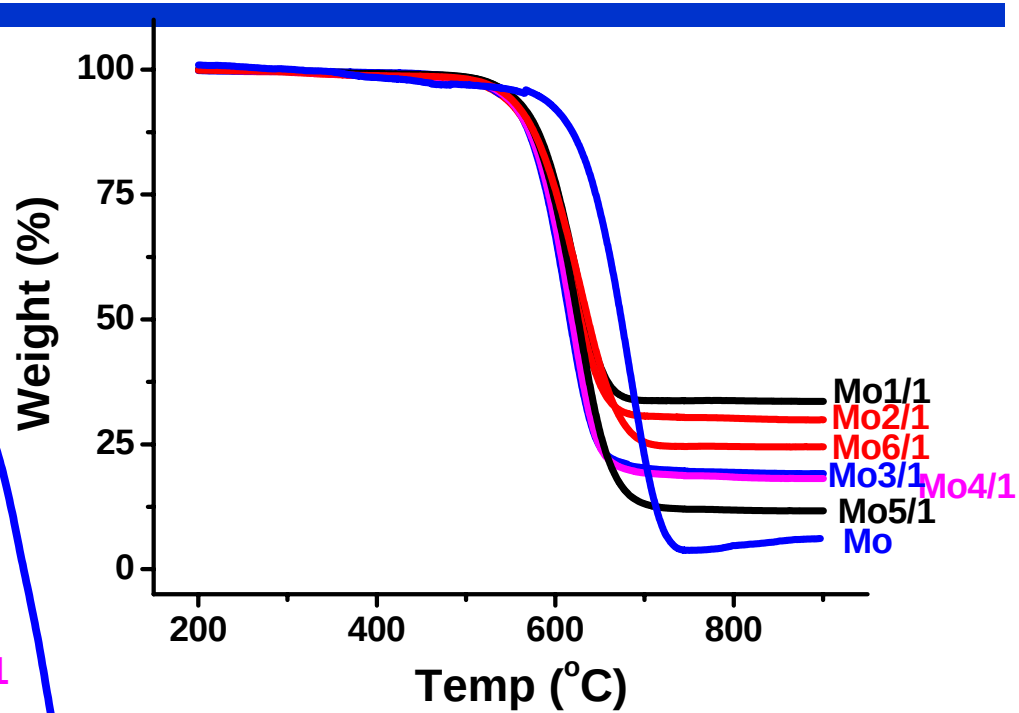
Mo Catalyst



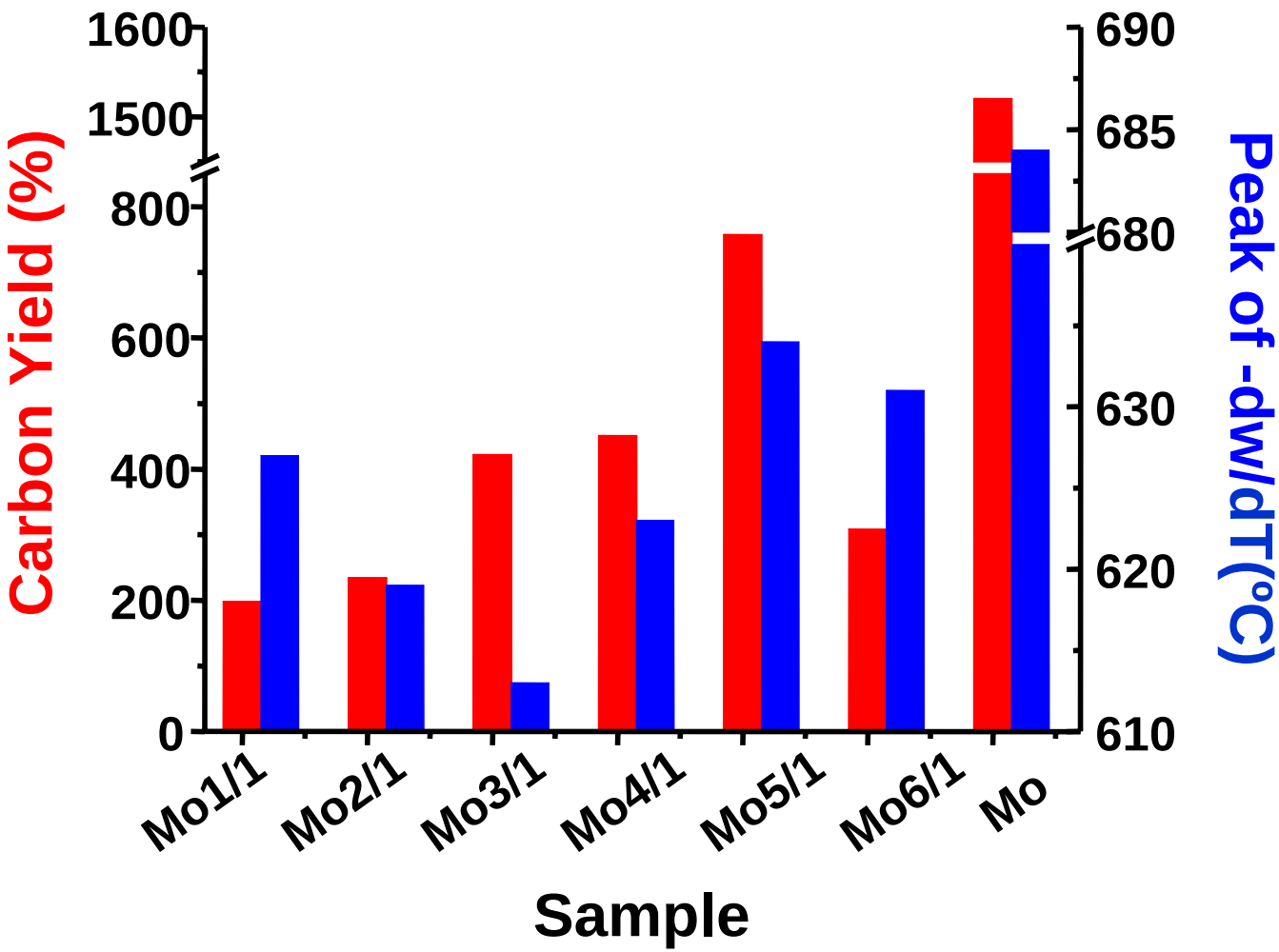
Effect of Catalyst Composite (18)

Mo Series

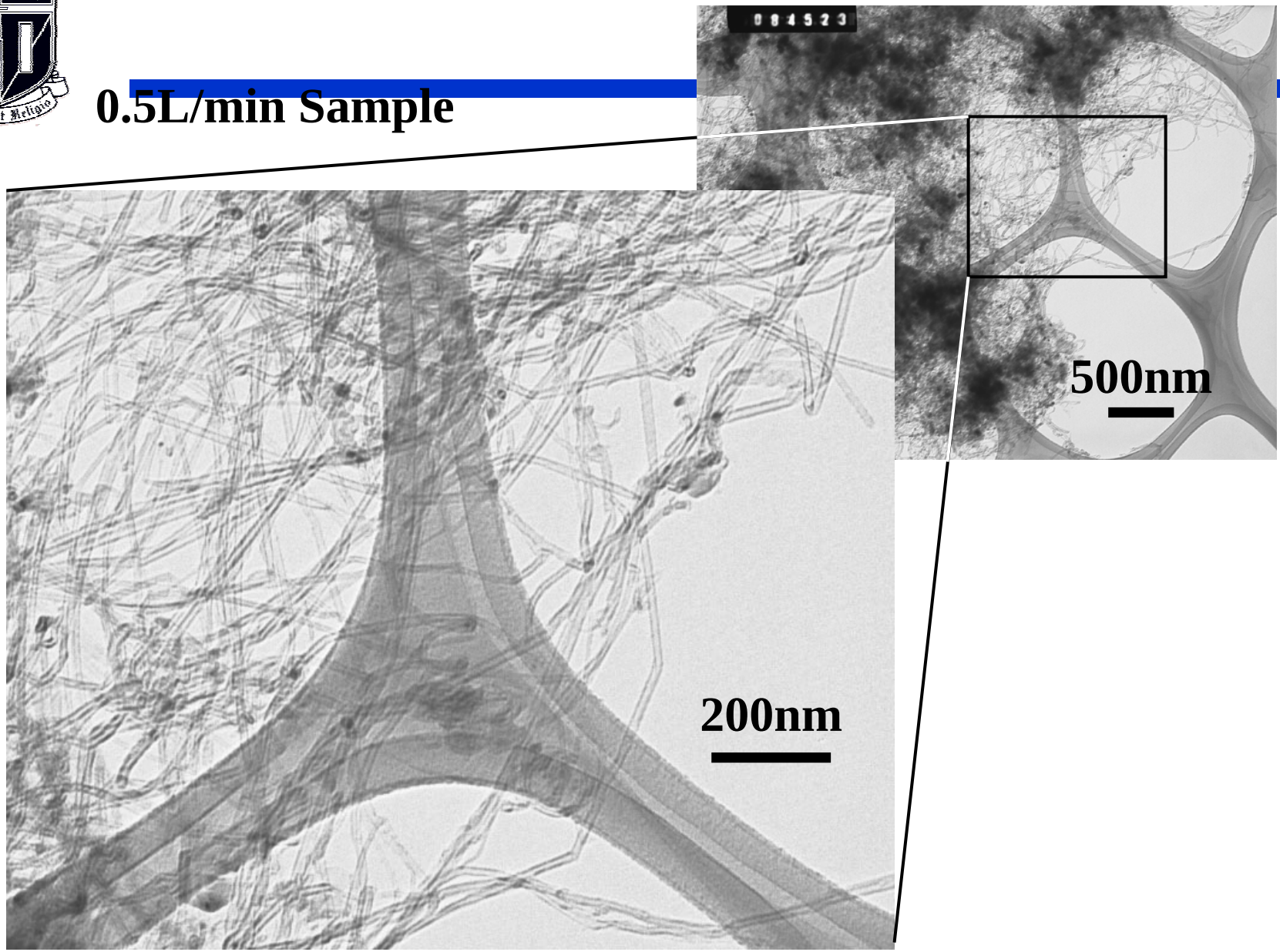




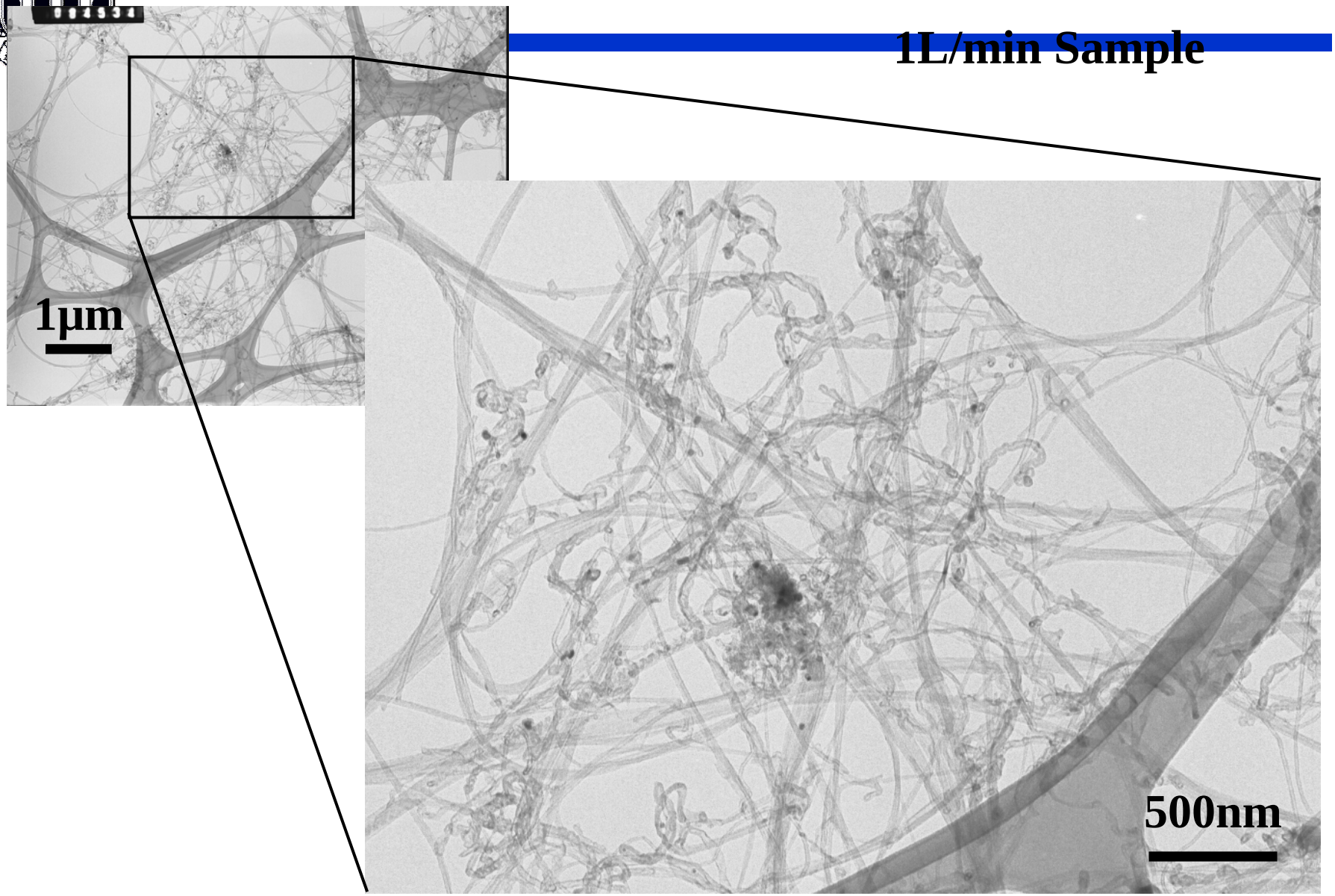
Effect of Catalyst Composite (19) ——— Mo Series



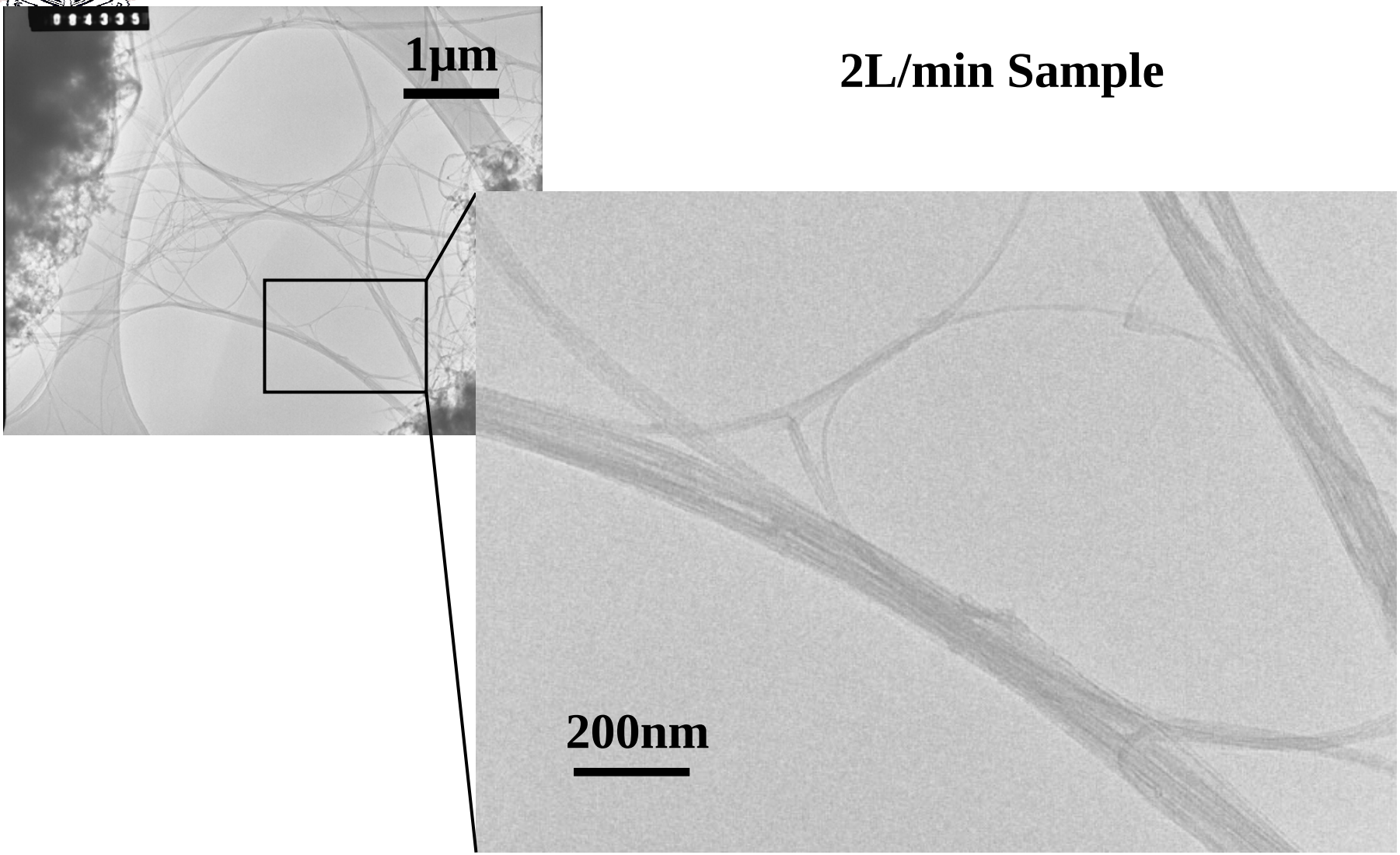
0.5L/min Sample



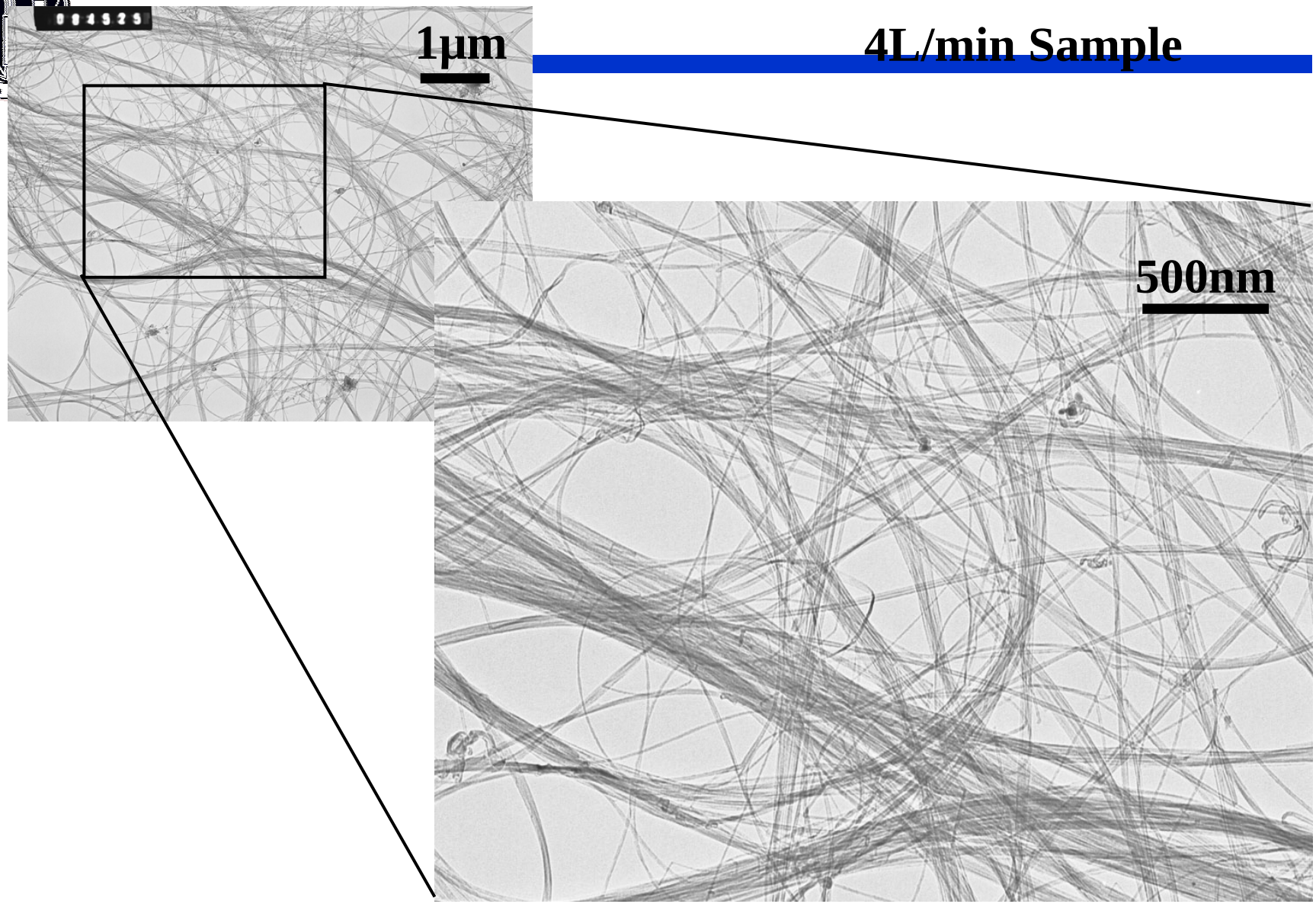
1L/min Sample

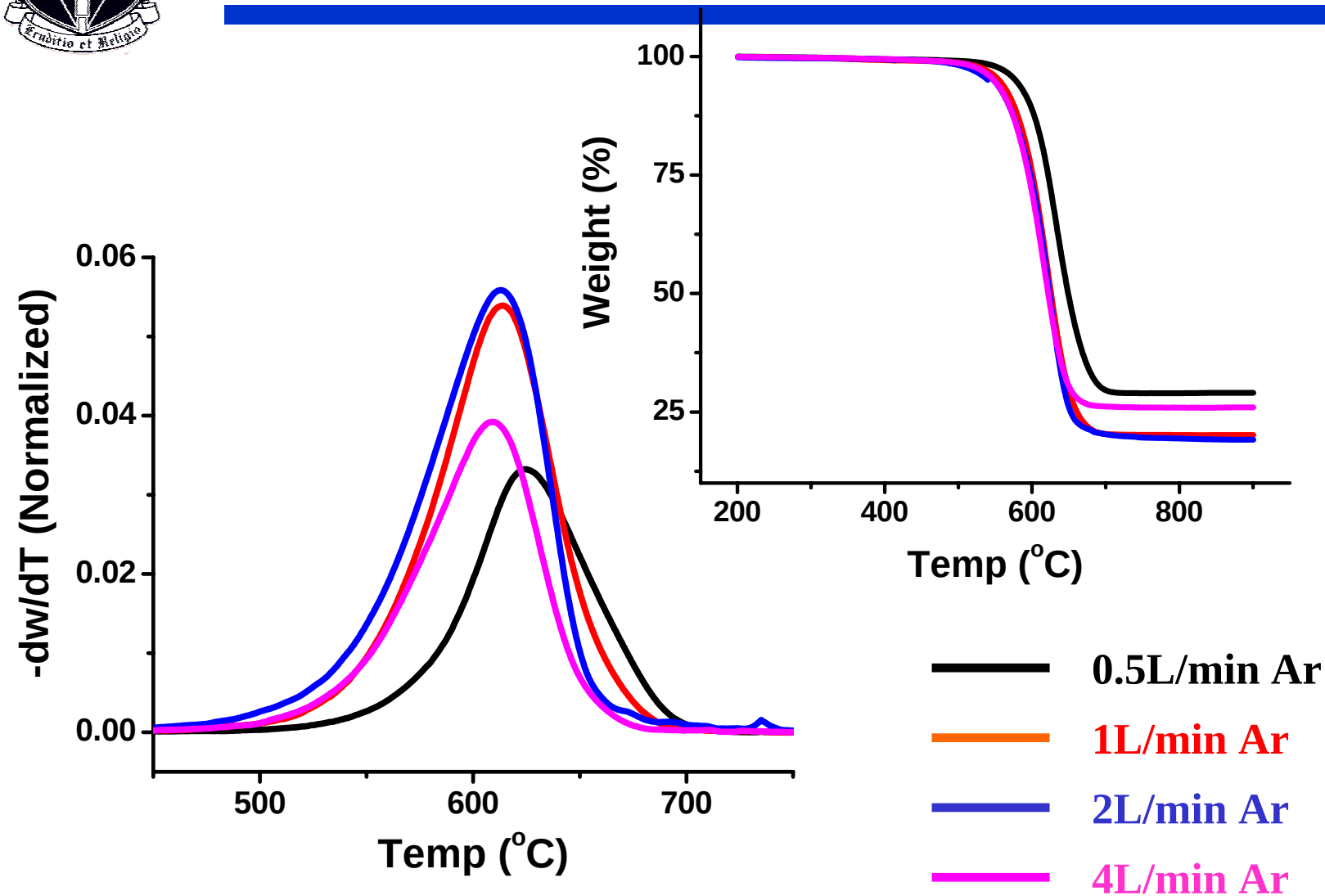


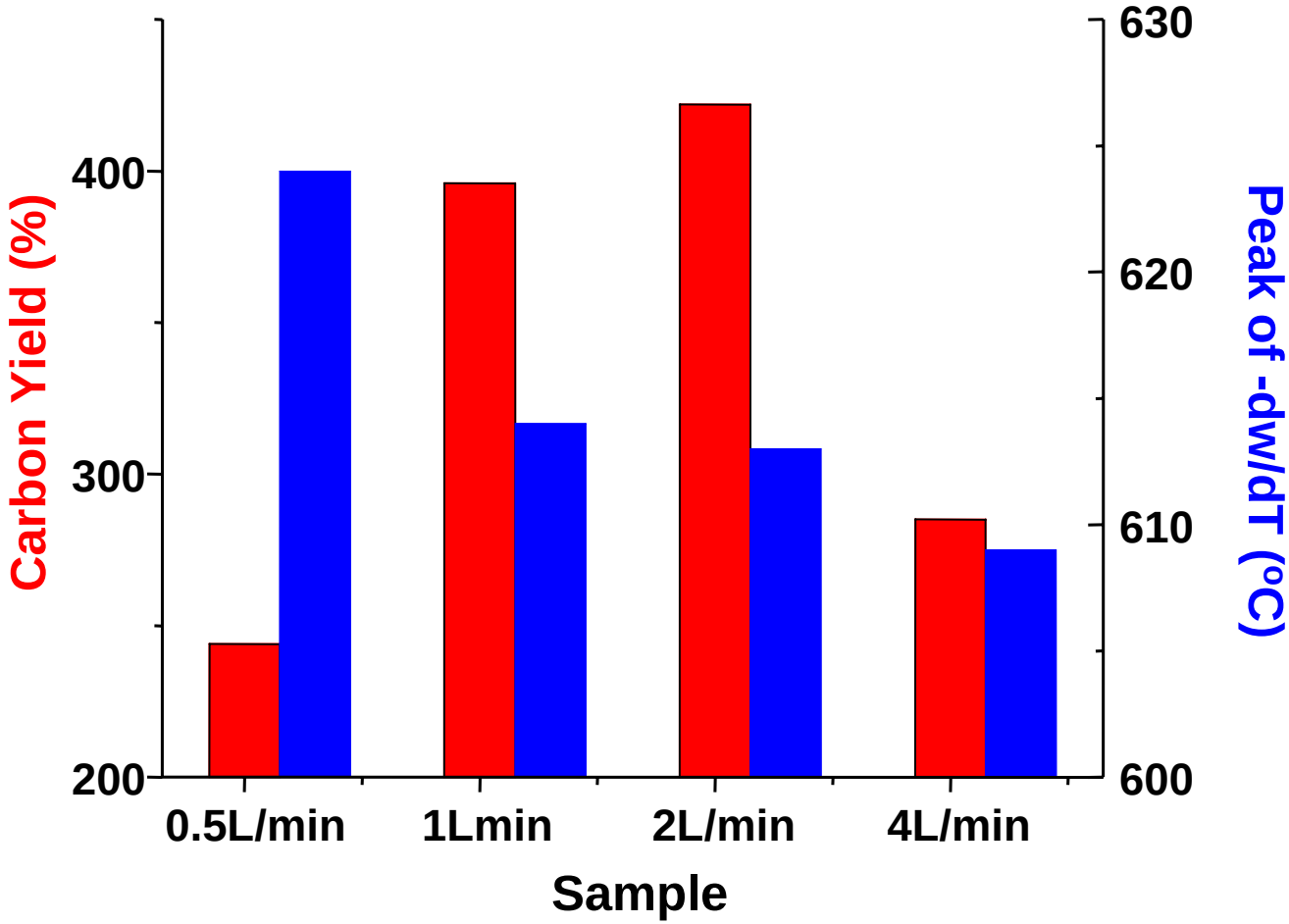
2L/min Sample



4L/min Sample

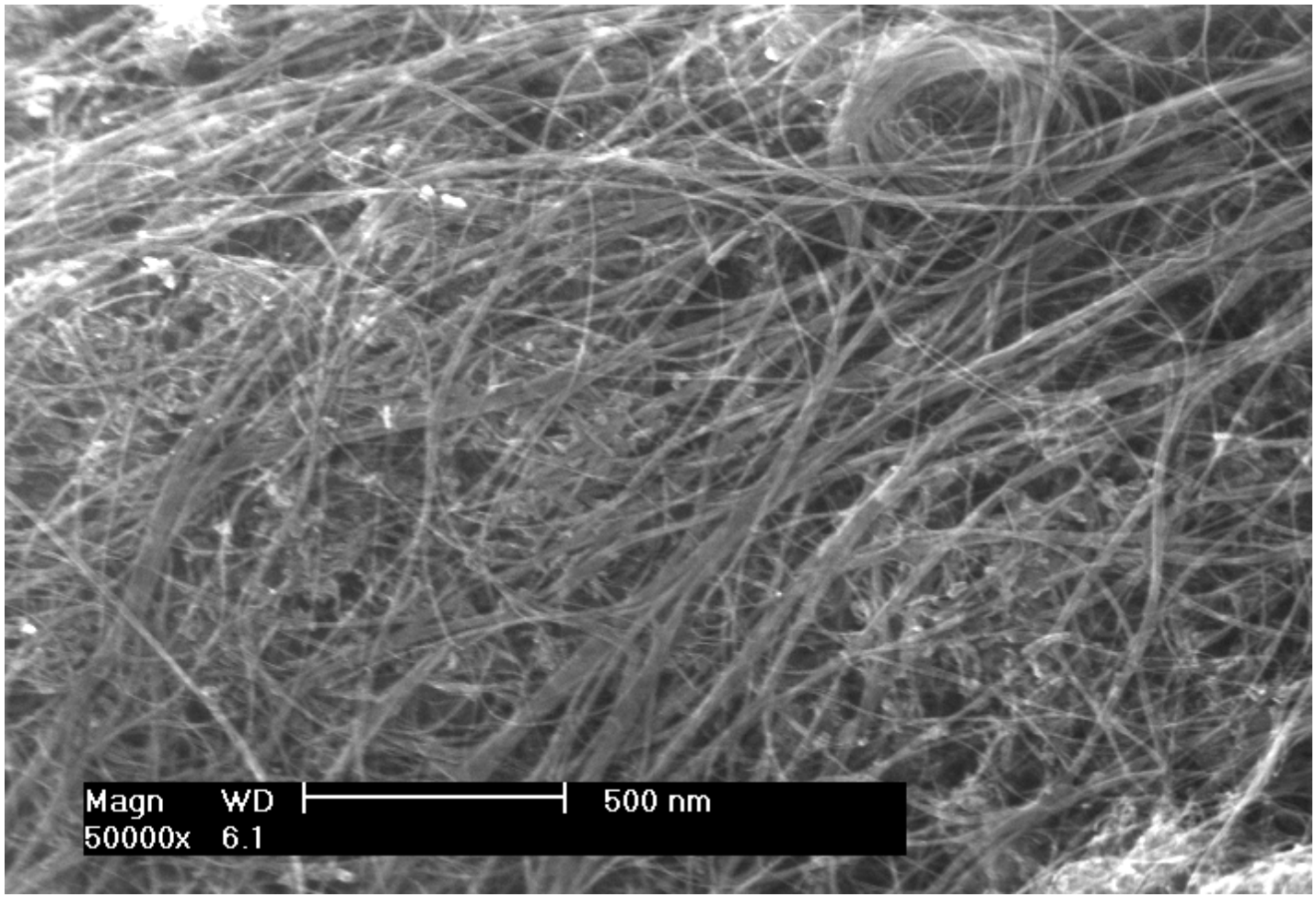



-dw/dT (Normalized)
0.06
0.04
0.02
0.00
500
600
700
Temp (°C)
Weight (%)
100
75
50
25
200
400
600
800
Temp (°C)
0.5L/min Ar
1L/min Ar
2L/min Ar
4L/min Ar

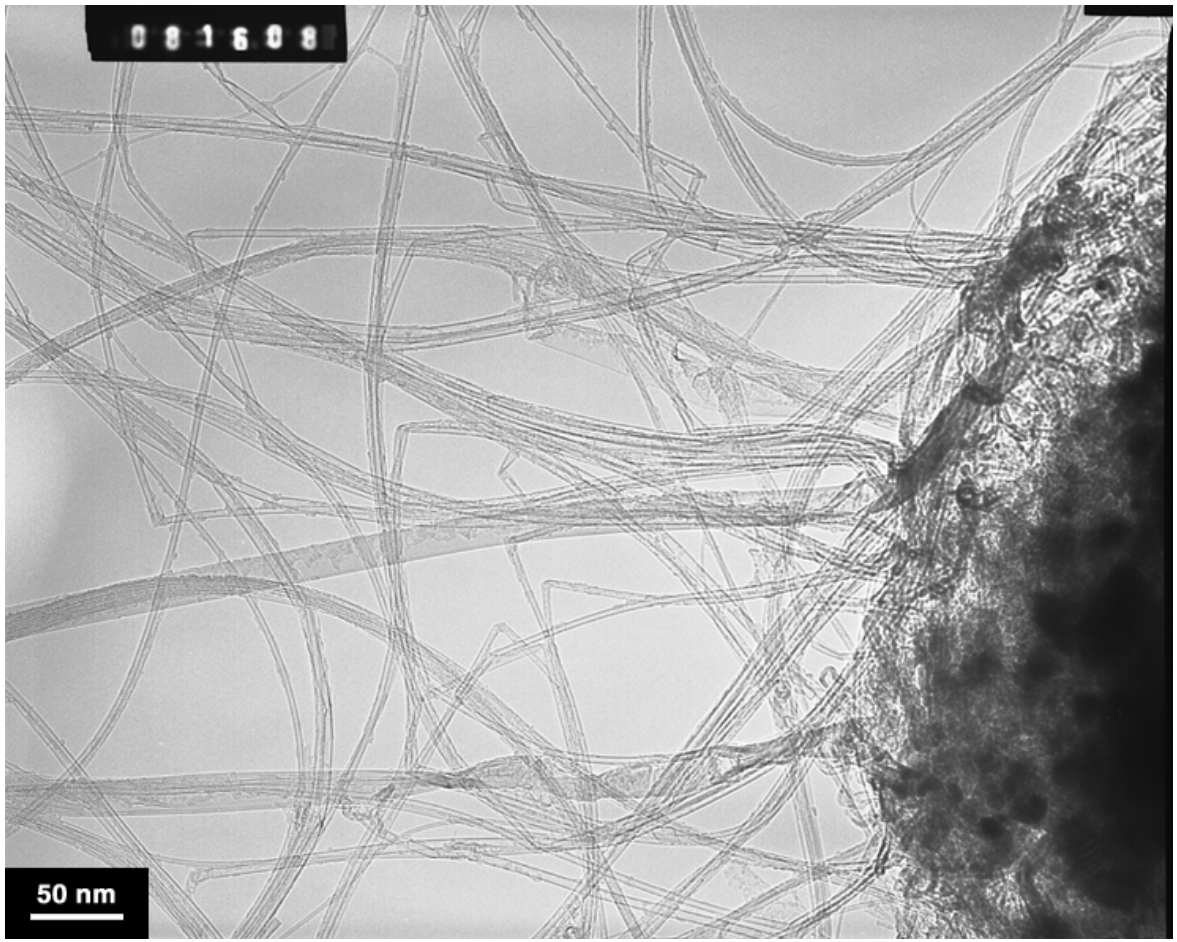

Carbon Yield (%)
400
300
200
630
620
610
600
Peak of -dw/dT (°C)
0.5L/min
1Lmin
2L/min
4L/min
Sample

Purification

Raw FWNT samples



Raw FWNTs



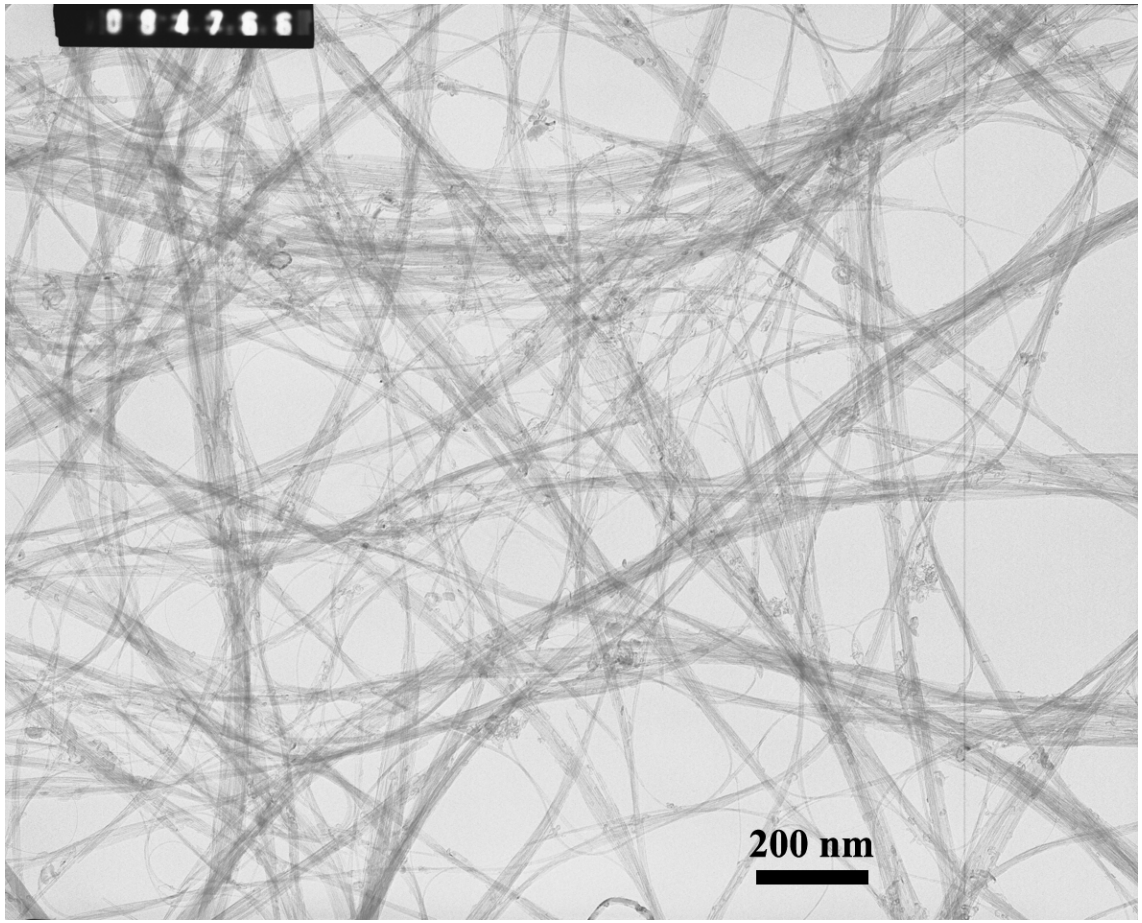
Composition of the Raw FWNT

- Support
- Support coated by Amorphous carbon & graphite sheets
- Catalyst
- FWNT
- Short MWNT & carbon fibers

Problems

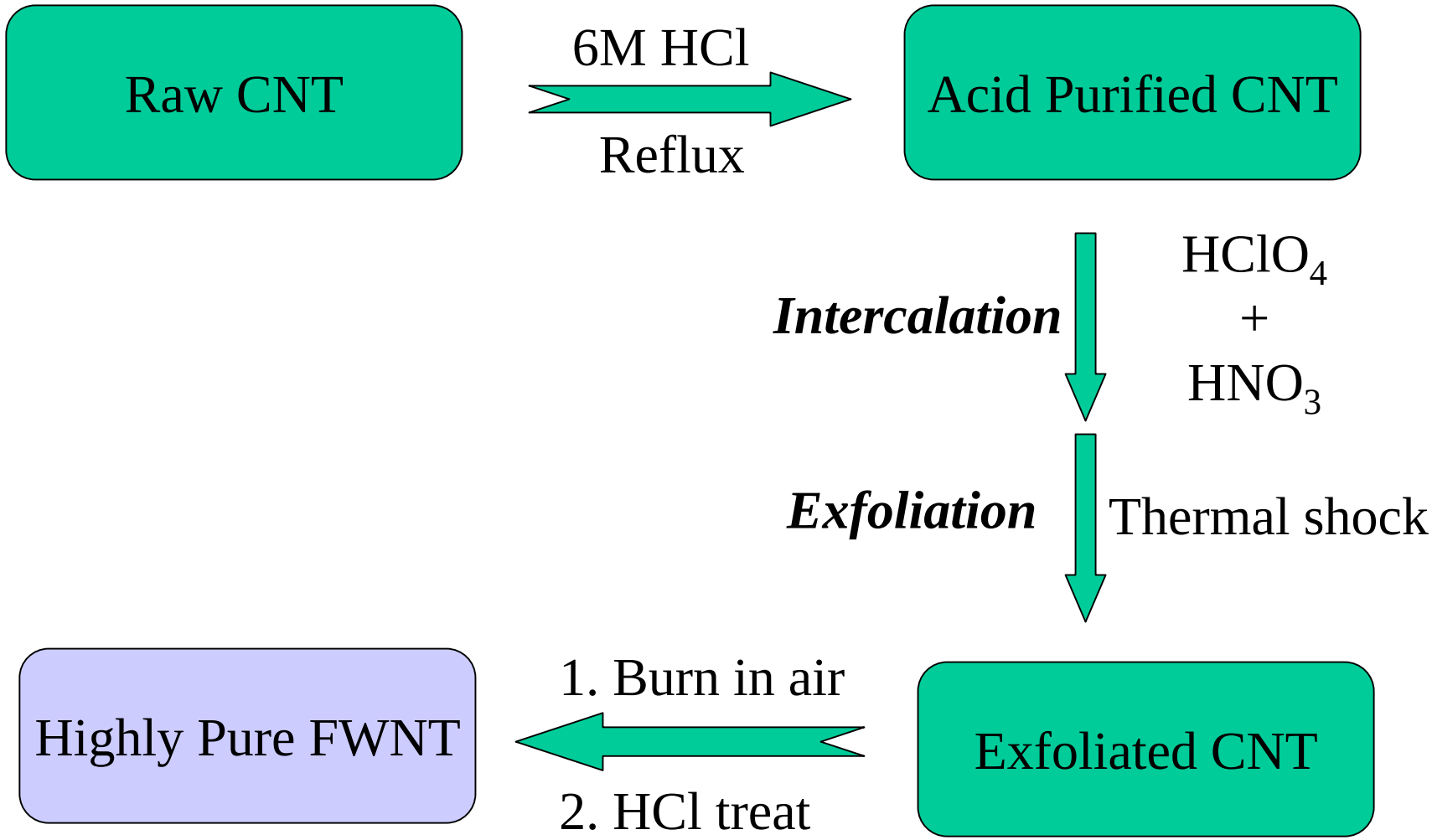
- Reflux in acid can only treat parts of support and catalysts. Support and catalysts coated by carbon species can not be removed.
- The temperature to burn the graphite species, MWNT and carbon fibers are similar or even higher than the temperature to burn FWNT. Also, these impurities are hard to be removed by physical separation method, especially MWNT and carbon fibers.

Purified materials following Rice Method for SWNTs

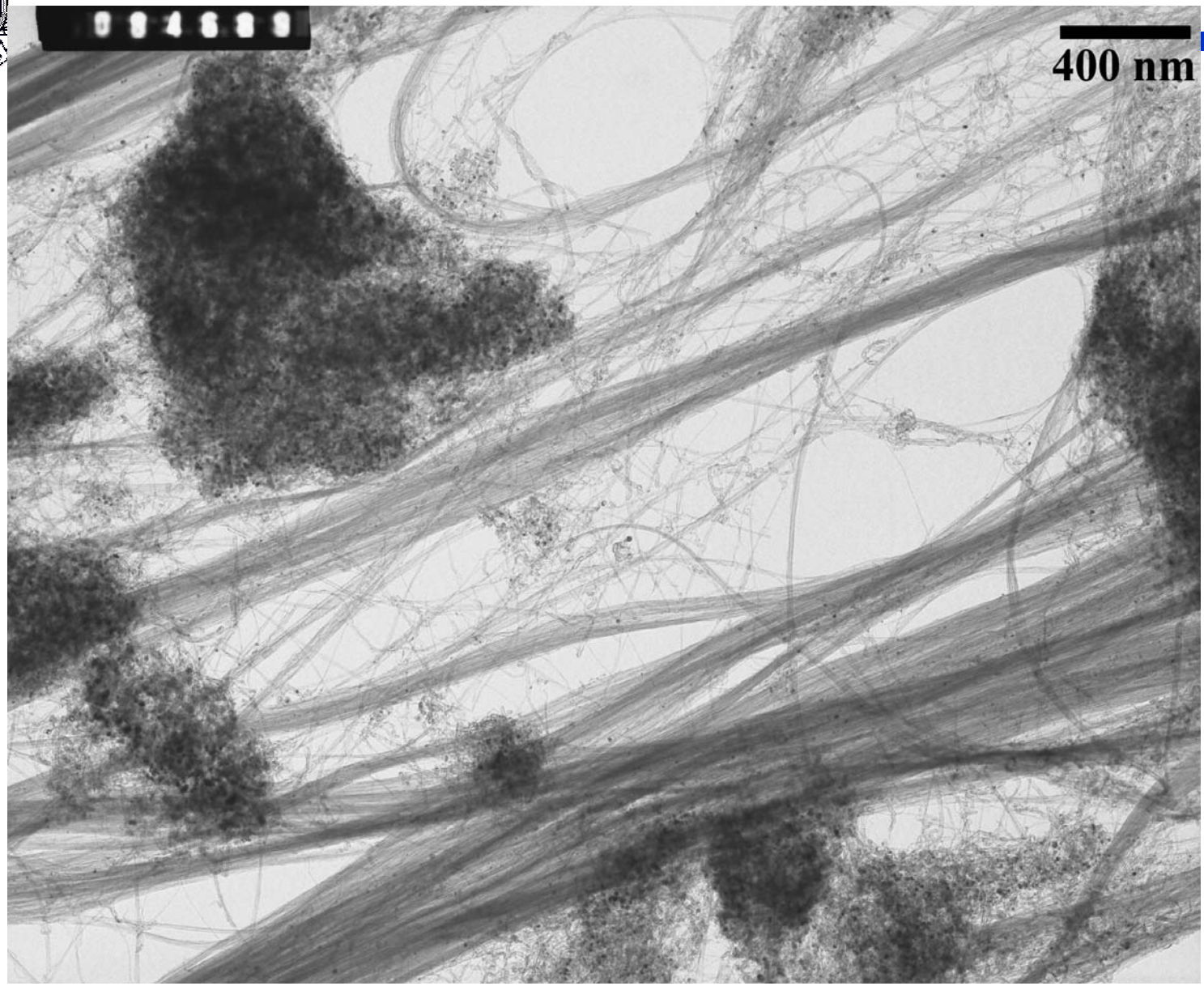


Low Yield

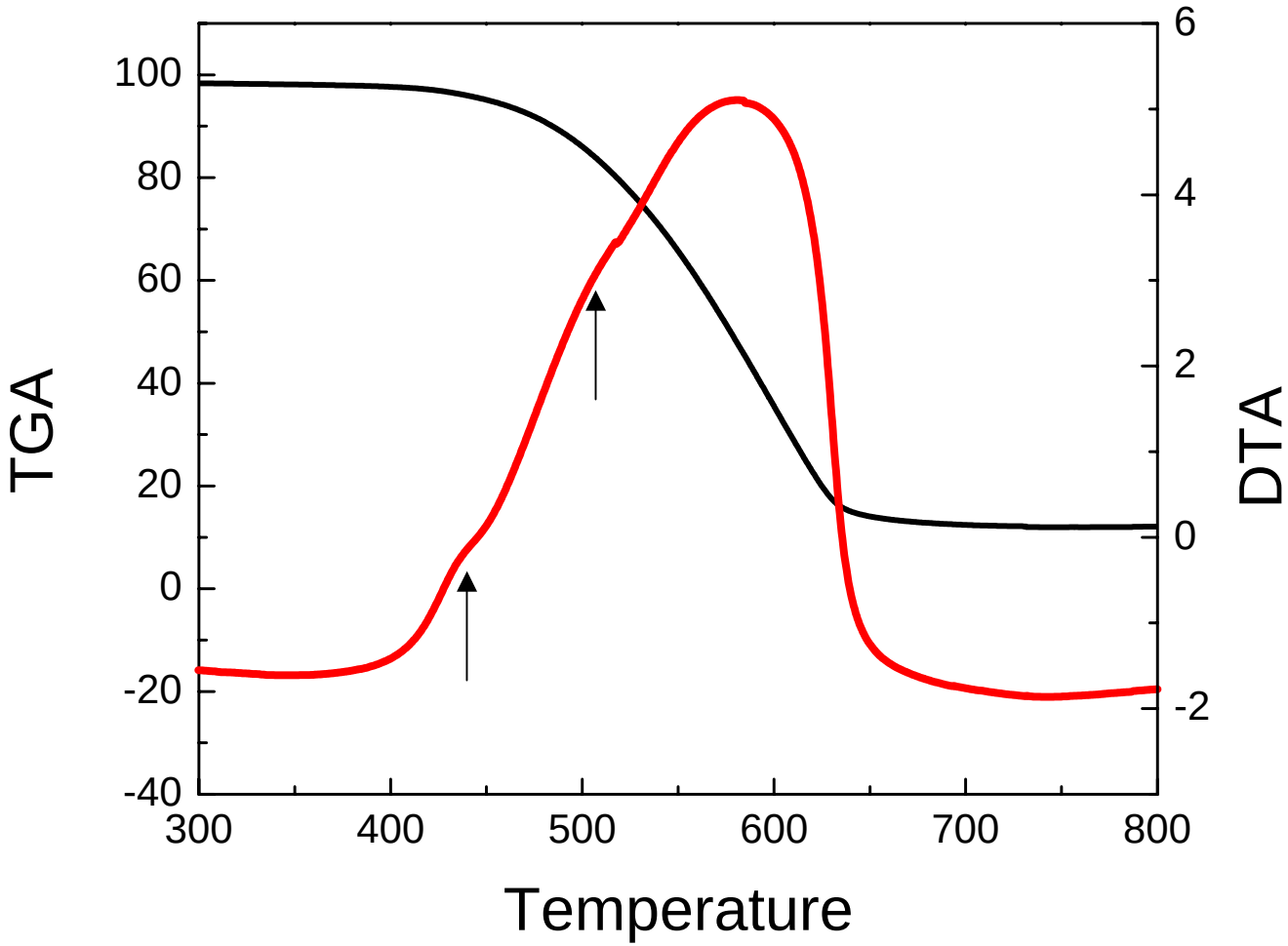
Exfoliation



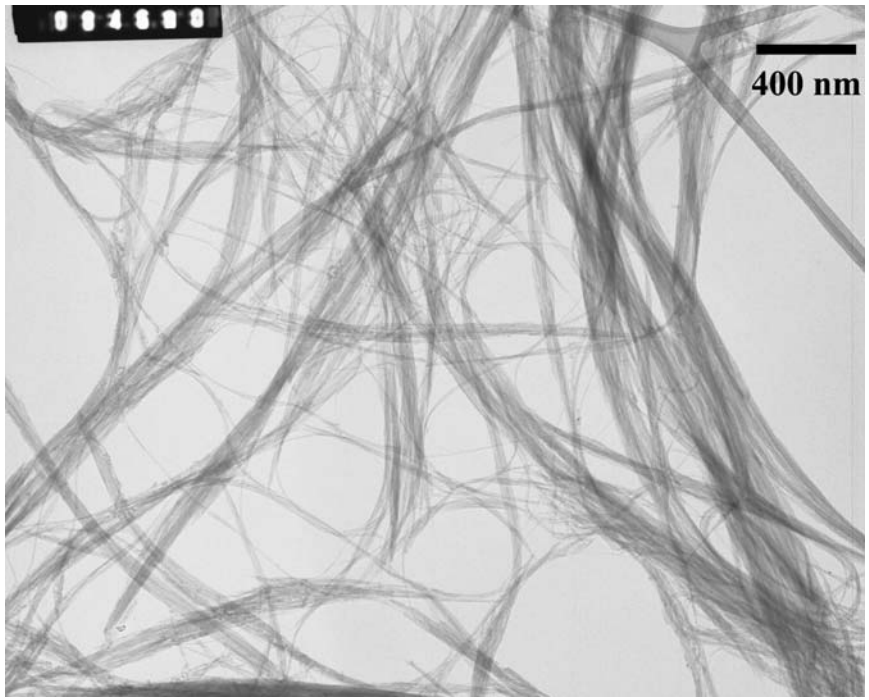
TEM Images of Exfoliated CNT

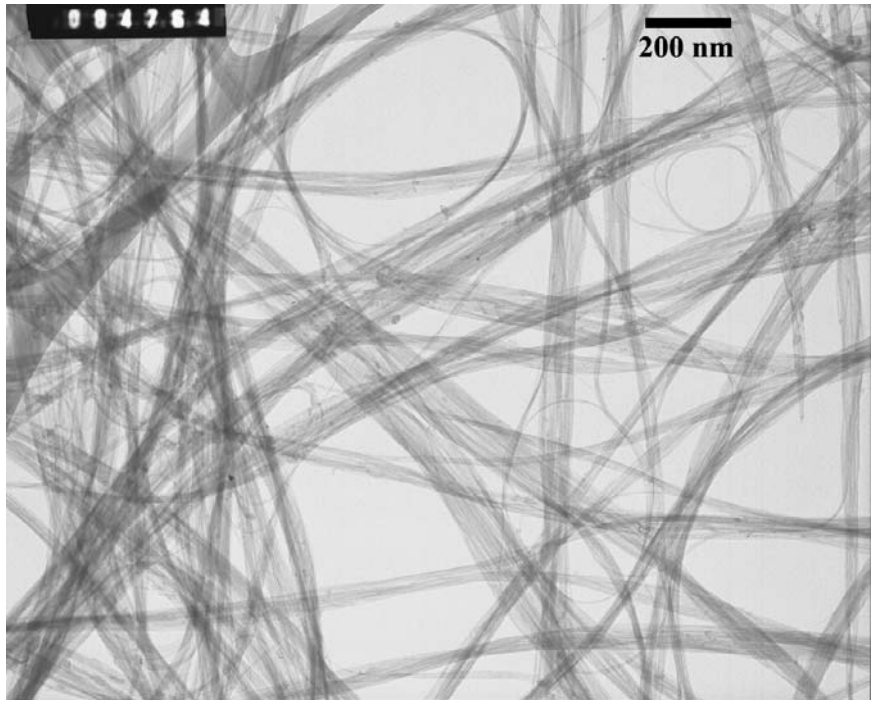


TGA of Exfoliated CNT



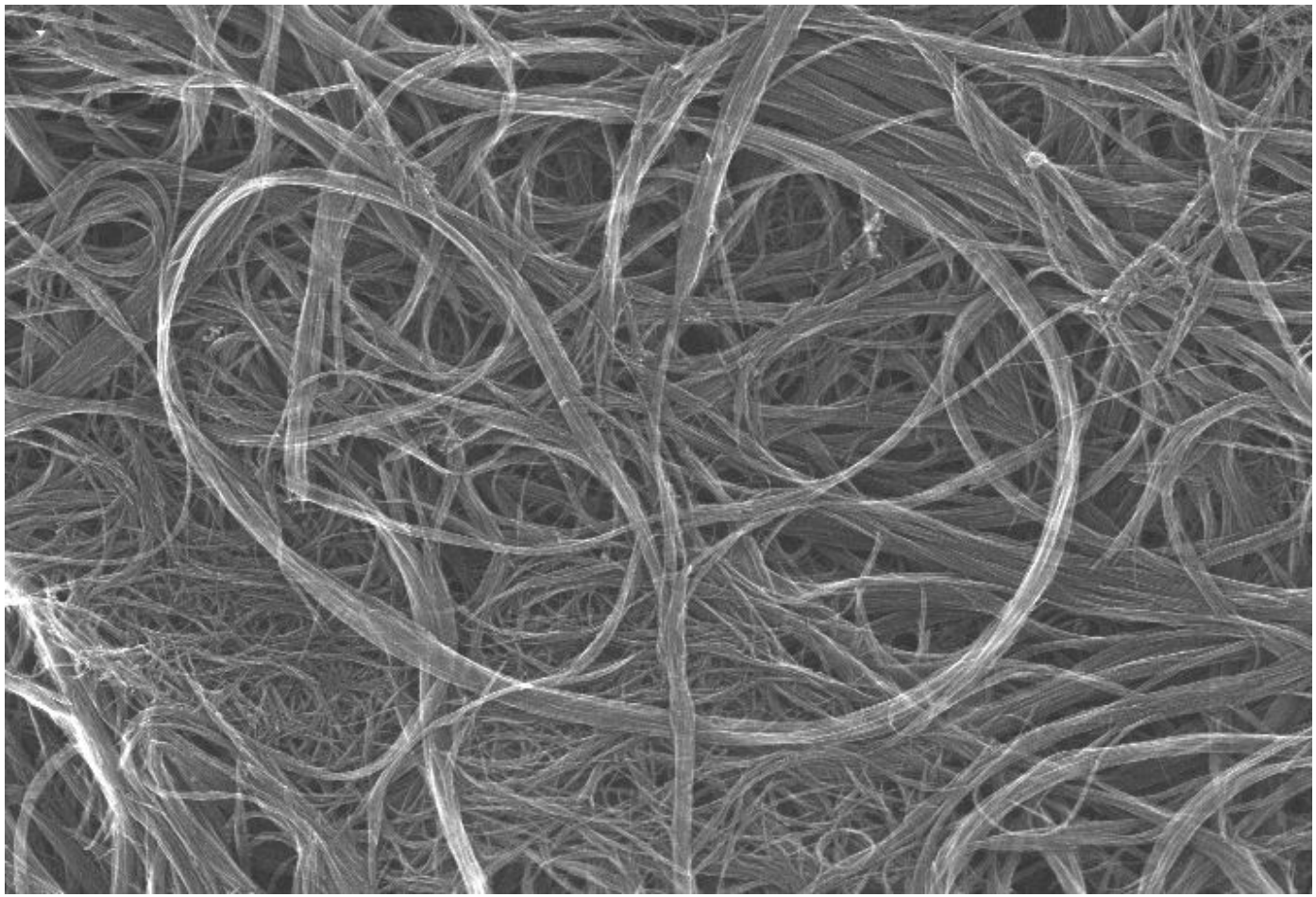
TEM Images of Highly Pure FWNT





Reasonable Yield ~ 20%

SEM Image of Highly Pure FWNT



Summary

- Small diameter and few-walled carbon nanotubes (FWNTs) have unique properties that make them suitable for bulk applications.
- FWNTs can be made in large quantity using CVD
- The field emission property of FWNTs is as good as or better than laser-grown SWNTs.
- TGA can be used to quickly evaluate the raw materials to determine their quality
- Purification yield can be significantly improved by adding an exfoliation step.

Acknowledgements

- Mr. Qian Cheng
- Mr. Hang Qi
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- Professor Lu-Chang Qin (UNC)
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- Dr. Qi Qiu (Xintek Inc.)
- NASA