Low-Z, Chemically Resistant, Microfabricated Carbon Composite

Transmission Electron Microscope Grids

Kyle Zufelt

A senior thesis submitted to the faculty of
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Bachelor of Science

Robert Davis, Advisor

Department of Physics and Astronomy

Brigham Young University

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ABSTRACT

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An issue that often impacts x-ray and electron analysis of electron microscopy samples is the presence of high-Z atoms in the chosen substrate. In many cases, it is also desirable that the chosen substrate be resistant to chemicals and various processing methods. We present an all-carbon transmission electron microscope (TEM) grid made by carbon nanotube templated microfabrication (CNT-M). Several membranes were deposited on the grids, including Formvar, amorphous carbon, silicon dioxide, and alumina. These grids provide a significant advantage in analytical TEM applications due to the absence of high-Z atoms and the improved chemical resistivity which allows for a wider range of sample preparation and processing techniques.

Keywords: TEM Grid, CNT-M

ACKNOWLEDGMENTS

I would like to express thanks to my parents, Amber and Lyle Zufelt, without whose help and support I would not have focused enough as a child to make it to college. I would like to thank Ed Stanfield and Ken Walker, who helped me to get interested in science as a hobby. I would like to thank Clayton Bingham for convincing me that a career in research was more beneficial than the career in medicine I was originally planning for. I would like to thank my wife, Anneliese Zufelt, for supporting me in my education.

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Chapter 1

Introduction

1.1 Transmission Electron Microscopy

A transmission electron microscope (TEM) is a microscope capable of resolving features too small to be seen using an optical microscope. A TEM takes advantage of the de Broglie wavelength,

$$\lambda = \frac{h}{p} \approx \frac{h}{\sqrt{2mV}}, \tag{1.1}$$

which tells us that all objects have a wavelength which varies inversely as mass and velocity. In a TEM, electrons (rest mass .5 MeV/c^2) are accelerated by a potential of up to hundreds of KeV, such that their wavelength, and thus the limit of their resolving power, is

$$\lambda \approx \frac{1.240 \cdot 10^{-6} eV \cdot m}{\sqrt{2 \cdot .5 \cdot 10^{6} eV \cdot 10^{5} eV}} = 3.92 * 10^{-12}\text{meters} = 3.92\,\text{picometers}, \tag{1.2}$$

or approximately 6 times smaller than the radius of a hydrogen atom. Thus a TEM has the potential to allows scientists to observe individual atoms in a sample.

The first TEMs were built in the 1930's, and since that time there have been many improvements in the design and capabilities of commercial TEMs. While the electron beam in a high

Figure 1.1 A Tecnai F20 Analytical STEM, similar to the TEM used to analyze our grids.

quality TEM can resolve individual atoms, it does not benefit a user if the electrons are absorbed by the sample before they can be observed. For this reason, samples that are prepared for observation and characterization in a TEM must be electron transparent and thus need to either be polished or deposited onto thin grids to achieve an acceptable thickness (<200 nm). At or below this thickness, the electron beam will interact with the sample but still continue down the column to a phosphorescent screen or a CCD which will display an image of the sample.

One advantage of TEM is a result of the interactions between the electron beam and the atoms in the sample. Electrons are charged particles, and accelerating a charged particle can cause it to radiate a photon. As electrons strike the atoms in a sample, the electrons in the sample move between different energy states. Each atom has characteristic energy levels given by the equation

$$E = \frac{-hcRZ^2}{n^2} \tag{1.3}$$

where R is the Rydberg constant ($10^7 \mathrm{m}^{-1}$), Z is the atomic number or number of protons per nucleus, and n is the principal quantum number representing the electron shell ($n = 1, 2, 3$, etc.). Thus by measuring the energy of emitted photons, a technique known as energy dispersive x-ray spectroscopy (EDX), we can determine the atomic makeup of a sample. Another analytical technique used in TEM research is electron energy loss spectroscopy (EELS), which measures the change in momentum of the electrons as they pass through the sample. Since the momentum of the electron can be changed not only by collisions with the atoms in the sample, but by phonon and plasmon excitations in an atomic lattice, this technique can reveal information about the bonds and crystal structure of the sample.

Analysis by means of EDX or EELS is sensitive to high-Z atoms not only in the TEM but in the vicinity of the detector. Commercially available microscopy grids are often made of a range of different metals, which may create unwanted background signals during such analysis. Metal ions are also undesirable for applications involving prolonged exposure to chemicals or biological

samples, where metals can react with the sample or the materials used in preparing the sample. TEM grids have also been made from beryllium, diamond, and other low-Z atoms, but these grids can be expensive or toxic.

1.2 Carbon Nanotubes and Chemical Vapor Deposition

Carbon is a low-Z atom which can be fabricated into three dimensional geometries. Previous attempts to incorporate carbon as a material in TEM grids include: casting carbon fibers in a polyester mold [1], and coating metals in a carbon nanotube film [2]. The former are not chemically resistant, and the latter do not avoid the use of high-Z ions. The two methods which currently exist for fabricating high-aspect ratio, all-carbon devices of controlled geometries are: carbonization of SU-8 photo-resist [3], and carbon nanotube templated microfabrication (CNT-M) [4]. The carbonization process of SU-8 structures results in shrinkage of up to 80% of the original dimensions of a structure. This does not allow for accurate dimensional control, an important element in the construction of TEM grids, which must fit into a 3.05 mm diameter slot in the TEM stage. CNT-M is a process whereby three dimensional carbon nanotube structures can be grown from a patterned two-dimensional catalyst layer. This vertical growth process preserves the cross section of the original patterned catalyst, allowing the creation of high aspect ratio structures with remarkable dimensional control. Here we use CNT-M to fabricate TEM grids of controlled geometry which are chemically resistant and devoid of high-Z and metal ion contaminants.

Carbon nanotubes are single-layer sheets of graphitic (SP2 bonded) carbon atoms, or graphene, which have arranged themselves in a cylindrical "tube" structure. Carbon nanotubes have been created and observed for many years [5], but the popularization and "discovery" are generally credited to a publication in Nature by Sumio Ijima in 1991 [6]. Carbon nanotubes can be fabricated by several methods including arc discharge, laser ablation, and chemical vapor deposition (CVD).

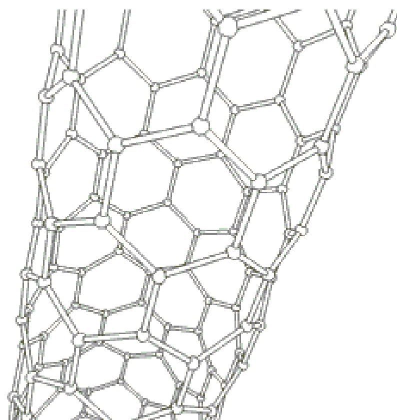


Figure 1.2 Diagram of a single-walled carbon nanotube.

The CNT-M process uses the CVD growth mechanism, which uses a catalyst material to enable carbon nanotube formation. Since the catalyst particle can be easily patterned, CVD allows carbon nanotube growth to be defined into nearly arbitrary geometries. CVD is also superior to other methods in terms of cost effectiveness.

1.3 Thin Films

As many TEM samples are prepared from materials or specimens smaller than the mesh size of the grids they are deposited on, it is often useful to attach a very thin (< 50 nm) film of some low-z material to act as a support for the deposited sample. Commercially available grids can be coated in carbon, various polymers, or various silicon compounds. The current methods for applying thin films to commercial grids are: a manual process of "floating" a thin film on the surface of a water bath and then attaching grids and allowing the films to dry; or a sensitive etch process to remove portions of a substrate and leave small areas of a suspended thin film. These thin films are very delicate, and it would be beneficial to develop a thin film deposition technique that requires as little manual handling and sensitive processing as possible, and which can process large batches

of grids simultaneously. A batch process for depositing suspended thin films is also of interest to basic science research investigating electrical and mechanical properties of thin films. More recently, TEM grids have been coated in small-grains of graphene, a single layer of crystalline carbon atoms. This represents the limit in thin carbon films. We coated our grids in several thin films, including graphene, amorphous carbon, silicon dioxide, aluminum oxide, and boron carbide. Our CNT-M process allows for batch deposition of thin films on TEM grids.

1.4 Lithography

Photolithography is a process for defining micron scale features analogous to taking a black and white picture. Photolithography allows one to use a transparent mask with specific areas coated in an opaque or reflective material to selectively expose areas of a photosensitive polymer, or photoresist, layer which has been deposited on a smooth substrate such as silicon. Depending on the type of photoresist used, either the exposed or the unexposed areas can then be dissolved in a developing solution. After development, a user can deposit thin films or etch away the exposed material. After processing, a more reactive chemical is used to remove the remaining photoresist. Because photolithography is often used to define micron scale features, users must be sensitive to the presence of dust and other particulate matter in the air, which are generally tens of microns in diameter or larger. To ensure quality results, photolithography is generally performed in a clean room, where the air is constantly filtered and circulated to maintain a very low particle count. BYU maintains a clean room at class 10, where the class rating is equivalent to the number of particles larger than 500 nm in diameter per cubic meter of air. By comparison, ambient air contains up to tens of millions of particles in an equivalent volume.

Chapter 2

Materials and Methods

2.1 Carbon Nanotube Grids

Carbon nanotubes were synthesized by thermal chemical vapor deposition (CVD), where carbon nanotubes were grown on prepared <100> silicon wafers by the decomposition of ethylene, a carbon-containing gas. The prepared wafers were patterned with a 30 nm Al_2O_3 diffusion barrier and a 7 nm Fe catalyst layer using standard photolithography techniques and lift off. CNT growth and infiltration was done in a Lindberg/Blue M 1" tube furnace. After a heating time of 10 minutes in hydrogen (200 sccm) to reduce the iron catalyst layer, CNTs were grown at 750°C with ethylene (150 sccm) and hydrogen (400 sccm) gases. CNT forests were infiltrated with carbon at 900°C by flowing 100 sccm ethylene and 200 sccm argon or hydrogen. The carbon filling ratio of a CNT-M structure has been previously shown to increase as a function of infiltration time [7]. The furnace was allowed to cool for 15 minutes while flowing 250 sccm argon, at which time the furnace was opened, with argon continuing to flow through the sealed tube, until the furnace temperature dropped to 300°C. The tube was then opened and the samples removed. Samples were etched in an Anelva RIE DEM-451 with a 300 W oxygen plasma, with 1.4 sccm of oxygen gas maintained

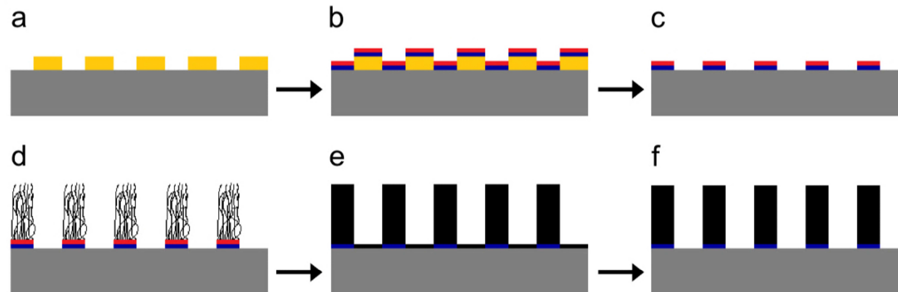


Figure 2.1 Overview of the CNT-M process. a - Use standard photolithography techniques to pattern a silicon wafer, or other desired growth substrate, with a negative of the desired growth cross section; b - Coat patterned wafer in a 30 nm alumina layer (diffusion barrier) and a 7 nm iron layer (growth catalyst); c - Lift off residual photoresist; d - Grow carbon nanotubes by CVD pyrolysis of ethylene; e - Infiltrate carbon nanotube forests with carbon, or other materials as desired; f - Plasma clean surface of growth substrate to remove "floor layer" (from infiltration)

at 100 mTorr for 5 minutes, to remove the floor layer that was deposited during infiltration.

2.2 Thin Films

A 0.5 % solution of Formvar powder (SPI Supplies CAS#63450-15-7) was prepared in 1,2-Dichloroethane as per manufacturer recommended instructions. Formvar was sprayed onto TEM grids, still attached to the silicon substrate, from a Sonotek Impact 120 ultrasonic atomizer. Formvar solution was dispensed at a rate of .500 mL/min as the raster head scanned over the sample 20 times at a rate of 1 inch/sec. Samples were allowed to cure at room temperature. Some uncured samples were exposed to steam to promote the formation of a "holey" Formvar film. Samples were placed in 49 % HF for 10 minutes to attack sample/substrate interface, rinsed in DI water for 10 minutes,

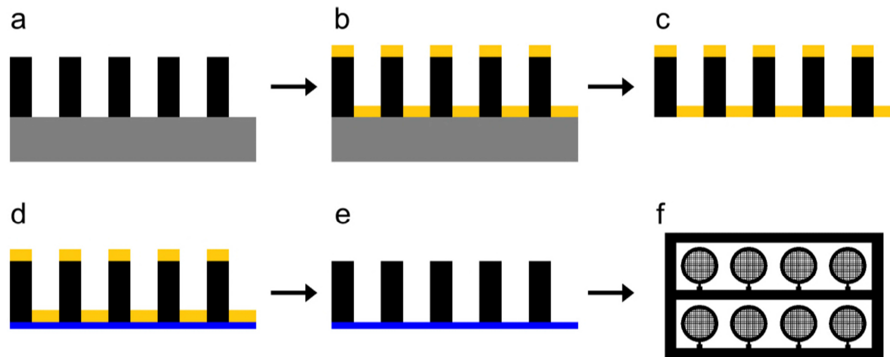


Figure 2.2 Overview of the thin film suspension process. a - Obtain CNT TEM grids (see above); b - Spray coat nanotubes and growth substrate in Formvar resin; c - Release CNT structures from growth substrate by HF etch; d - Deposit the desired thin film on the growth-substrate-defined Formvar surface; e - Anneal structures at 400 C in argon to remove Formvar; f - Grids are made in locked sets for easy handling

and removed from substrate by gentle exfoliation.

Carbon films were sputtered on Formvar-coated grids in a Kurt Lesker PVD-75 system. Graphene was grown as described in [8] and then transferred as described in [9] to TEM grids coated in "holey" Formvar. Silicon dioxide films were deposited in a PlasmaLab Model DP800 PECVD. Boron Carbide films were deposited in a custom magnetron sputter deposition system. Aluminum oxide films were deposited by a Denton Vacuum e-beam evaporation system. After thin film deposition, grids were annealed in argon at 400°C for 15 minutes to ash the Formvar layer (See Figure **??**).

2.3 Structural and Chemical Tests

Carbon nanotube cantilevers (200 μm wide by 2 mm long) were grown to a height of 100 μm and infiltrated with the same parameters as used for TEM grids. Cantilevers were force tested

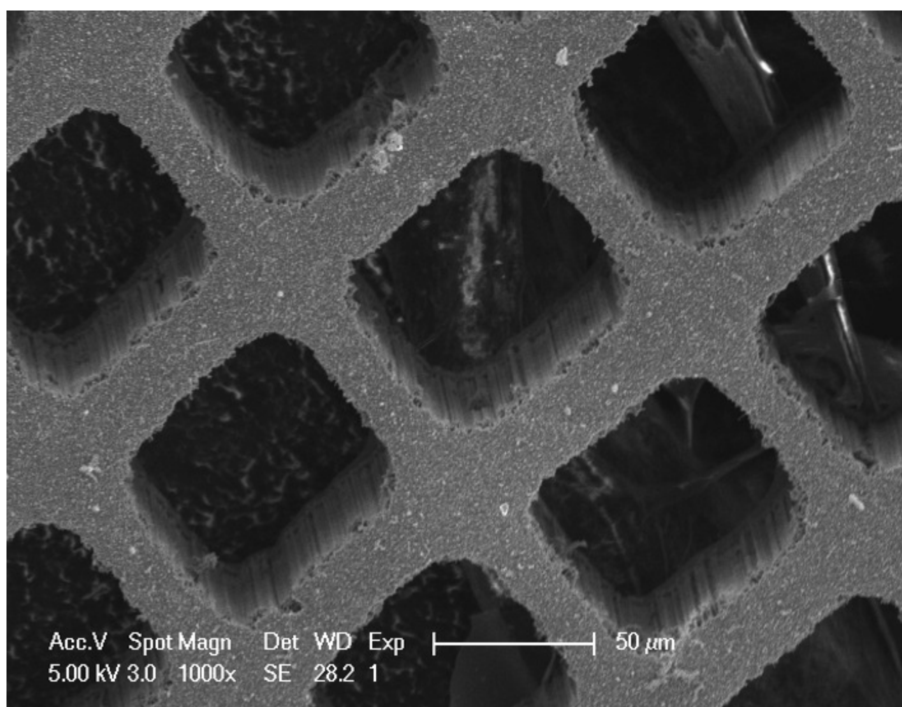


Figure 2.3 SEM image of a CNT-M TEM grid, tilted to emphasize vertical sidewall growth.

with an Instron 3342 tensile strength tester equipped with a 50N force transducer, which extended cantilevers at a rate of 0.5 mm/min until they broke. The strength tester measured the force applied and the deflection of the cantilever. Chemical testing was performed by placing grids in KOH, HCl, or HF solutions for several hours. Other grids were analyzed in a Rigaku Miniflex II XRD system with a Cu X-ray source. Raman spectra were collected at room temperature by using a PI-200 Raman Analyzer equipped with a TE-cooled DU401-FI Higher charge coupled detector and a 785.11 nm laser. EDX and EELS data were taken in a Tecnai F20 Analytical STEM. Thin films were deposited on glass slides and thickness confirmed by characterization with a Veeco Dimension 5 AFM and/or by ellipsometry.

Chapter 3

Results and Discussion

3.1 Results

Each 4" silicon wafer contained 250 individual TEM grids locked together in sets of 4. Figure 2.2 is an SEM image confirming the fabrication of grids 3 mm in diameter with mesh spacing of 200 or 300 holes per square inch. Grid height was a function of CNT growth time. Flowing hydrogen during carbon infiltration leaves CNT structures attached to substrate, while flowing argon causes spontaneous release. Analysis of cantilever testing by Euler - Bernoulli beam theory gives a Young's modulus of 7.0±0.5 GPa and a yield tensile strength of 128±11 MPa (See Figure 5), which agrees well with prior published values [7].

EDX analysis of grids showed carbon peaks with no other atomic signatures (Figure 3.6). This indicated the absence of the Fe growth catalyst and Al_2O_3 diffusion barrier used during the growth process. XRD analysis showed broad peaks for infiltrated CNT structures indicating a grain size on the order of 2 nm (Figure 3.4). Chemically etched grids showed no measurable deformation or change in mechanical properties of the grid. EDX detected adsorbed potassium ions from KOH etched grids though there was an absence of Cl and F ions from HCl and HF chemical tests,

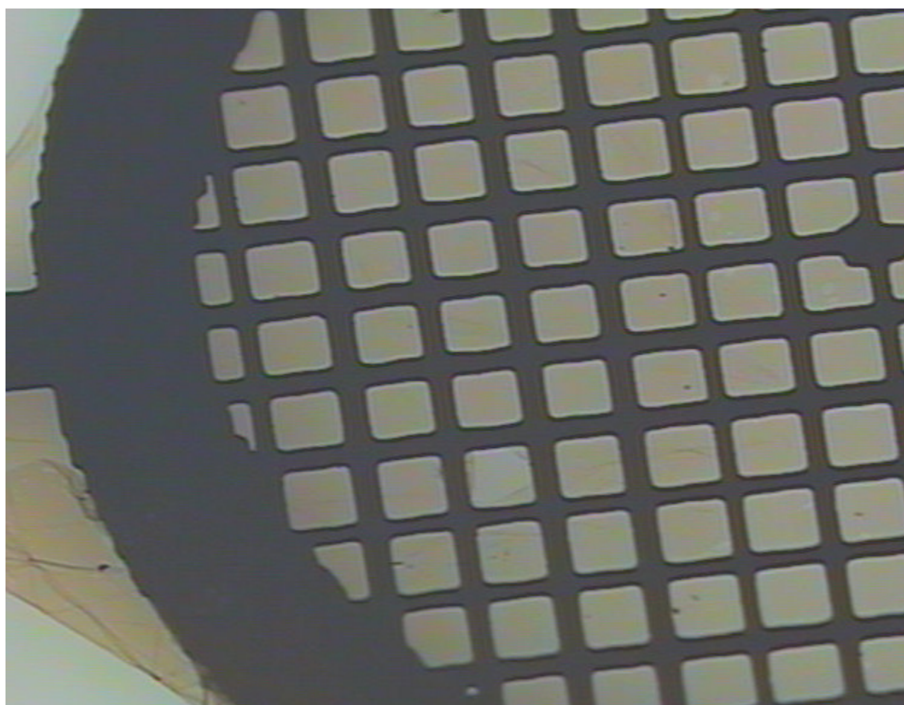


Figure 3.1 Optical micrograph of a 10 nm amorphous carbon film on a CNT-M TEM grid in locked geometry.

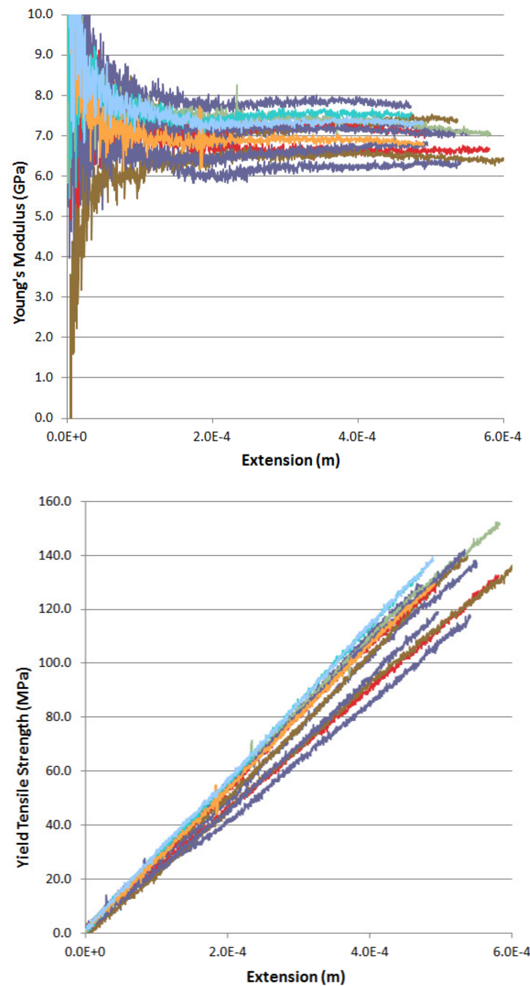


Figure 3.2 Graphs showing Young's modulus (left) and tensile stress (right) of 13 cantilever structures. The average of the data sets are 7.0±0.5 GPa and 128±11 MPa, respectively.

respectively (see Figure 3.6).

AFM measurement of Formvar films on glass slide shows a 30 nm film. AFM measurement of amorphous carbon films on silicon wafer shards show the thickness of the film increases as a function of exposure time in the PVD system. 15 minutes produced a 10 nm film, and 25 minutes produced a 40 nm film. Suspended thin films were optically confirmed to be present in the majority of the TEM grid pores. Various thin films were observed by TEM to have a highly amorphous

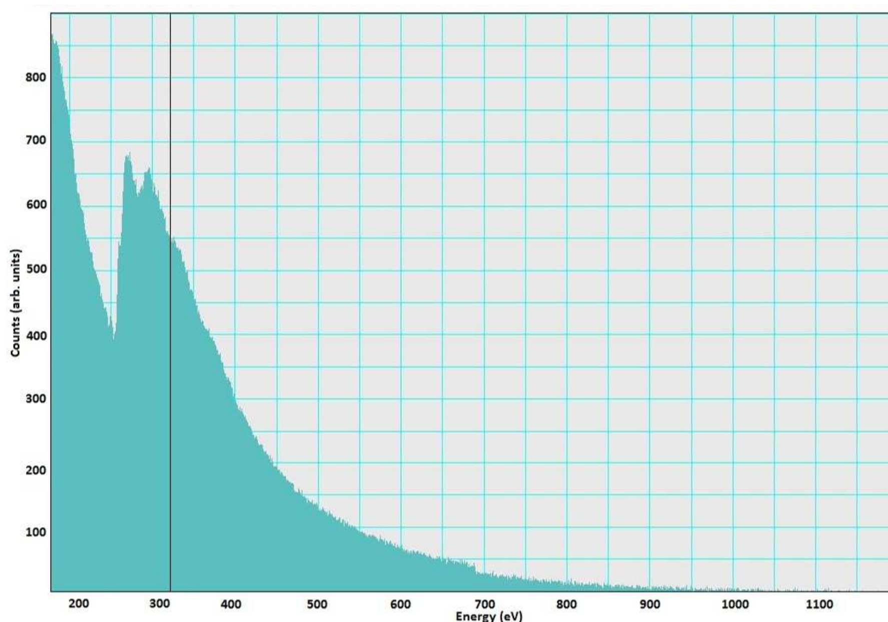


Figure 3.3 EELS data from a CNT-M TEM grid. Broadness of peaks indicates small grain graphitic bonding in carbon.

structure, desirable for providing a low-background support (See Figure 3.5).

EELS of carbon structure deposited during infiltration showed good agreement with an EELS library entry for nanocrystalline graphitic carbon(See Figure 3.3). EELS of sputtered carbon gives better agreement with library data for amorphous carbon. RAMAN of graphene matches data taken on graphene provided by researchers in the Paul McEuen group at Cornell University (See Figure 3.7). AFM measurement of glass slides coated in Formvar and annealed at 400 C show a small residue that is likely carbon.

3.2 Discussion

Carbon grids are more resistant to bending than metal grids. They are more chemically resistant than metal grids, and contain no detectable high-z atoms. The microfabrication process used

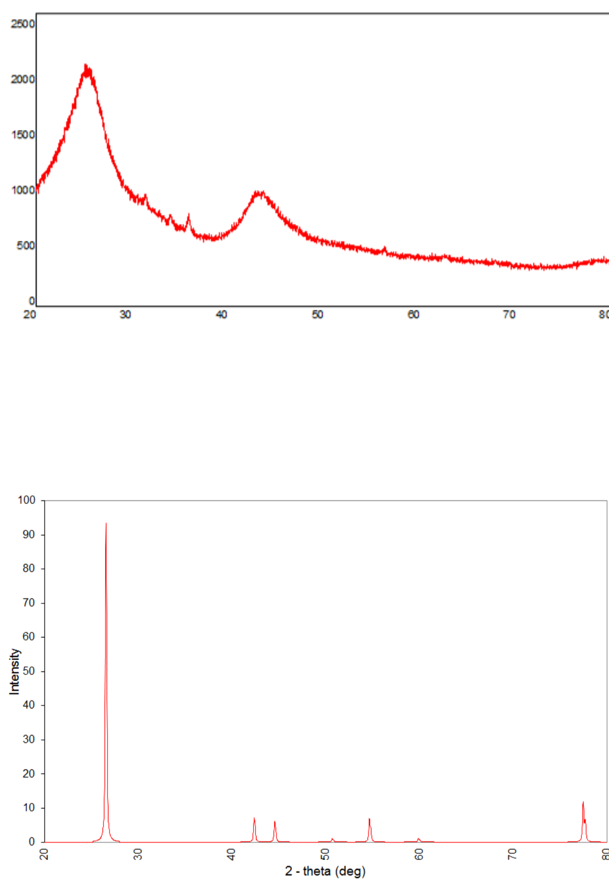


Figure 3.4 XRD data from [top] CNT TEM grids major peaks at 3.5 and 2.1 Å correspond to (002) and (100) peaks and [bottom] graphite powder.

to make the grids allows reliable control over the dimensions of the grid. The microfabrication process also allows control over the number of grids that are locked together during growth and processing. The broad peaks seen in XRD analysis are characteristic of a range of small crystalline grains, which is consistent with carbon nanotube structures.

Graphene has very high strength and generates very little contrast in the TEM, making it a good choice for use as a support film.

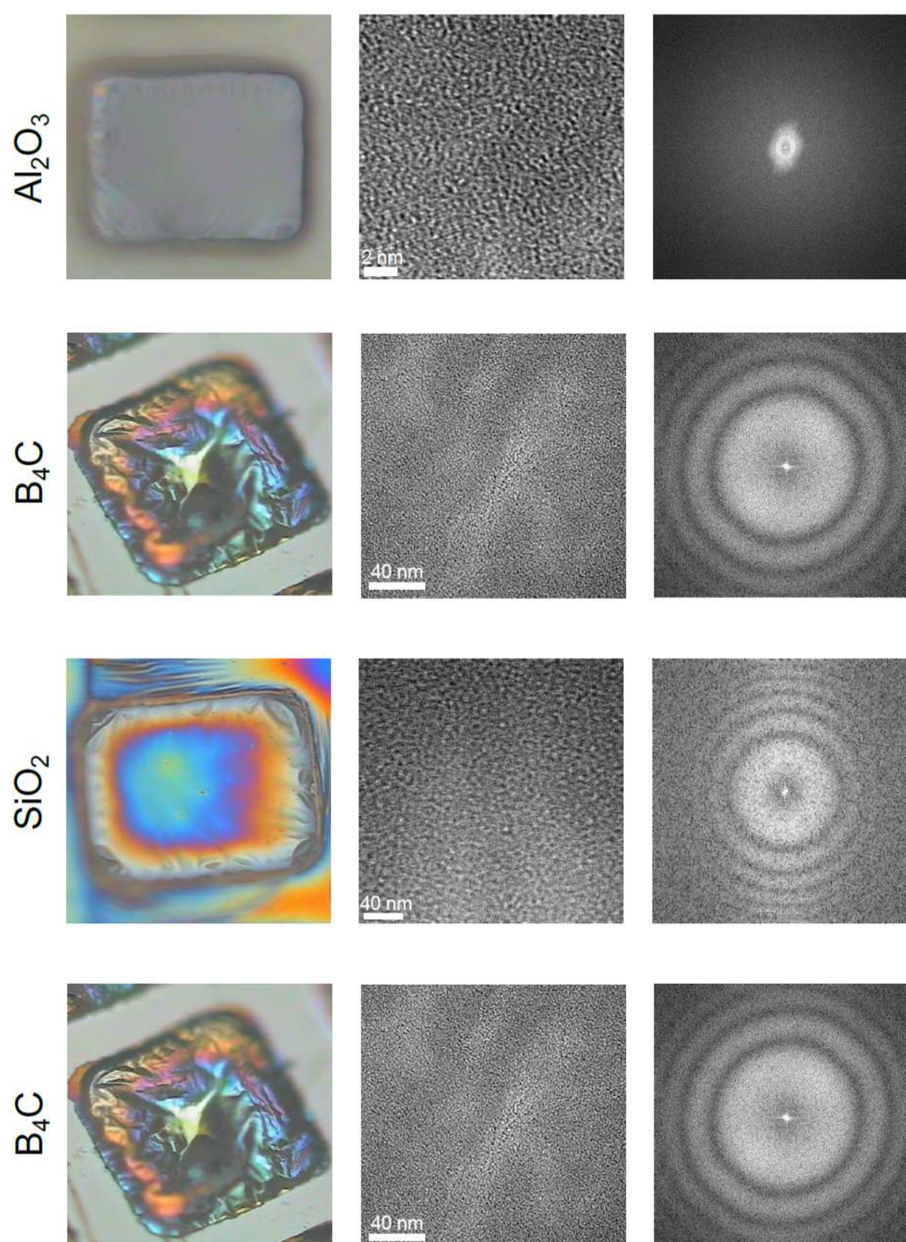


Figure 3.5 Our thin film suspension technique allows for flexibility in choice of thin fim materials, including (from top to bottom): 30 nm alumina, 25 nm boron carbide, 30 nm silicon dioxide, and 10 nm amorphous carbon thin films. Each row contains (from left to right): an optical microscope image, a TEM micrograph, and an FFT of the TEM micrograph. With the exception of alumina, which shows a crystal structure corresponding to the formation of β-alumina ($a = 2.38$) from sodium contamination, all films deposited by this process are highly amorphous.

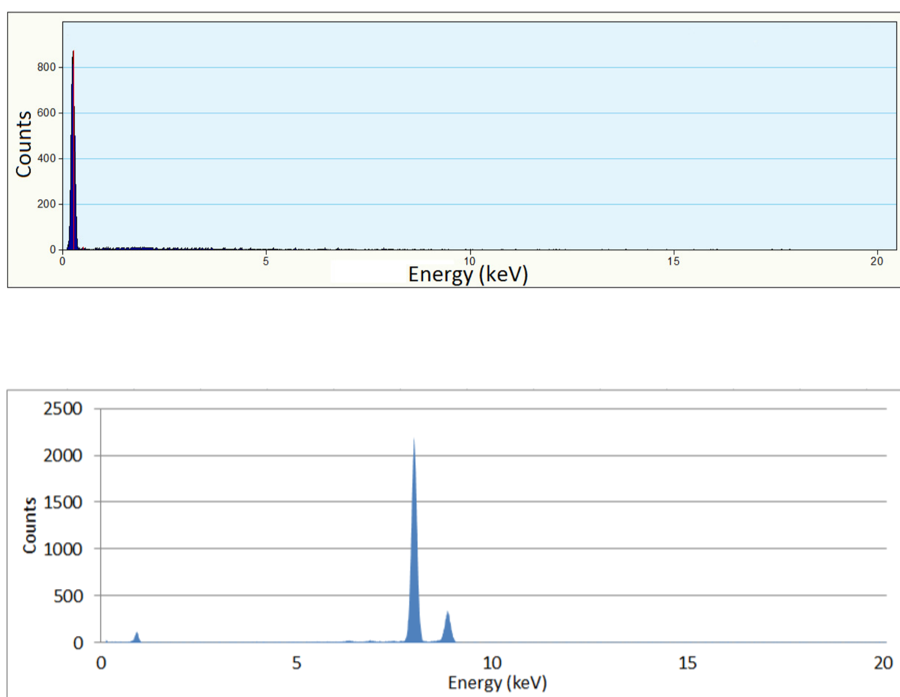


Figure 3.6 100 s of EDX data collected from all-carbon TEM grid (top), and a commercial copper grid (bottom). Carbon peak corresponds to Kα emission (0.227 eV), copper peaks correspond to Kα (8.040 eV), Kβ (8.905 eV), and Lα (1.023 eV) emission, respectively.

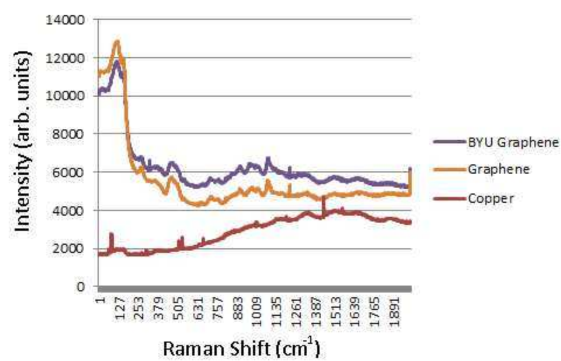


Figure 3.7 RAMAN of graphene.

Chapter 4

Conclusions and Future Directions

We have made TEM grids that are composed entirely of carbon. These grids are more resistant to bending or breaking than commercially available grids, have a greater tensile strength, and can be made and processed in batches. Grids have been coated in suspended Formvar, carbon, alumina, and boron carbide thin films.

Future work may include demonstrating the scalability of the processes involved in CNT-M TEM grid fabrication. It may also include extension of our thin film work, both in the variety of materials tested, and in applying our technique to other projects. Ideas that have been considered include studying CNT growth from a suspended alumina membrane, and investigating compatability of our process with other polymers.

4.1 Acknowledgements

I thank Stacey Smith for assistance with XRD analysis, and Jason Lund for Linear Beam Theory modeling. This work was funded in part by a BYU ORCA grant.

Appendix A

Thermal Evaporators

As part of my group responsibilities, I was placed in charge of maintenance and training for our two thermal evaporators. The first is an older system named "JIM," after Jim Thorne, an emeritus professor of chemistry at BYU who purchased the system. The other is a newer Denton evaporator acquired from the BYU biology department in 2011. What follows is an outline of the maintenance responsibilities for each system, followed by user instructions for each system. General vacuum maintenance will not be covered, as it is expected that the reader will be familiar with these principles from a physics lab course. Never hesitate to ask for help or further clarification from department staff if you are not sure what to do.

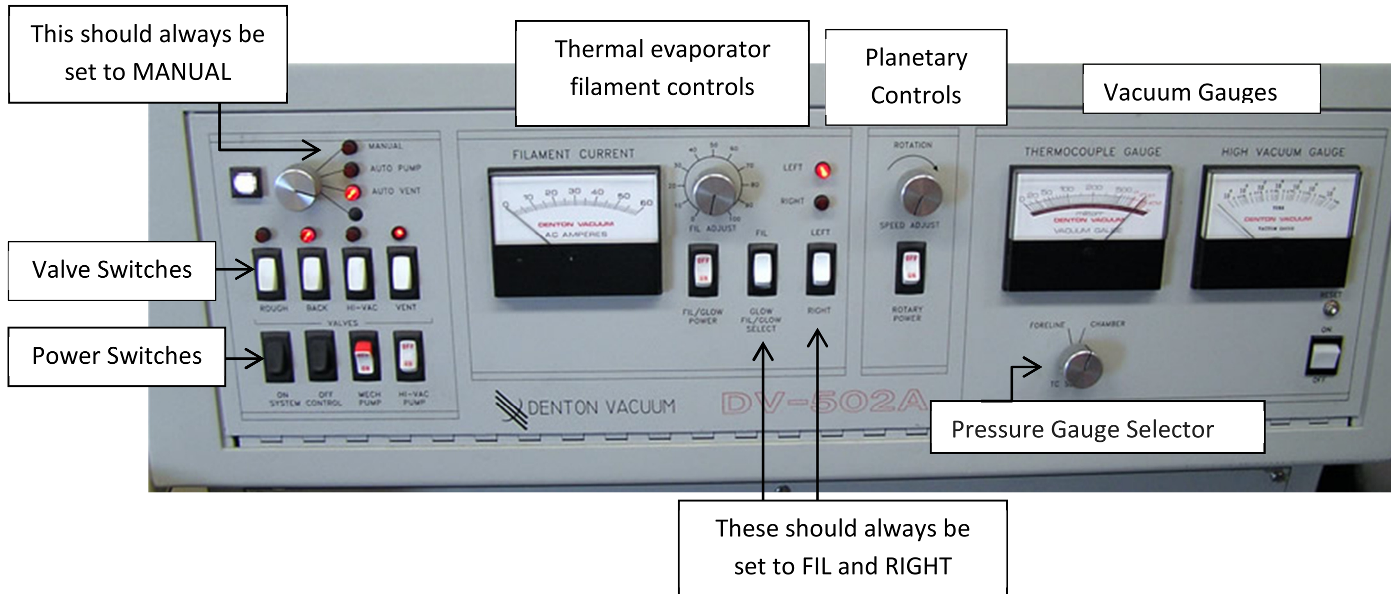
JIM is attached to a cryo pump that was acquired used, and thus has contaminants in the cryo head chamber. We attempted to remove these contaminants by an extensive purge with helium gas, but did not observe any improvement. Generally, the contaminants freeze out and donâĂŹt cause issues with normal operations of the system. However, the cryo pump needs to be regenerated periodically by the student with responsibility for JIM, and this refreezing process can lead to ice formation in the cryo head, which causes a very loud grinding noise and may cause damage to the cryo pump. When the cryo pump needs to be regenerated, or in other words when the cryo pump is no longer maintaining a temperature of less than 20 K, the cryo pump compressor must be shut off

and allowed to come to room temperature overnight. This causes many of the adsorbed molecules to desorb into the gas phase, increasing the pressure in the cryo chamber. When the cryo chamber has warmed up, it must then be pumped out by the rough pump for several hours to encourage outgassing of adsorbed molecules. When the cryo chamber has been sufficiently rough pumped down, the compressor may be turned on again. As the cryo head cools down, the gate valve should be closed to prevent damage due to the interaction between pumps. Stay with the system and listen for abnormal noises from the cryo head. If noises occur, the regenerative process must be repeated, starting by shutting off the compressor. Eventually, usually after two or three attempts, the contaminants will freeze out in a non-essential location and the cryo pump will resume normal operations.

The Denton evaporator is attached to an oil diffusion pump backed by a system roughing pump. To prevent oil back-streaming into the chamber, the system uses a cold trap that is cooled by liquid nitrogen. The shutter in the system is controlled by two screws in a solenoid valve towards the back of the system. Each screw controls one direction of motion, and the direction assigned to "open" and "close" can be reversed by switching the compressed air lines at the piston on the front of the chamber. The screws are very sensitive, small rotations lead to large changes in the speed of actuation. We have been fortunate enough to not need to perform significant maintenance on the Denton as of this time, and so we leave it to future students to gain more experience in troubleshooting the system.

Denton Thermal Evaporator – Standard Operating Procedure v1.1

12/19/11 – Kyle Zufelt



The Denton system has three electrodes for thermal evaporation and sputtering. It is not currently plumbed for sputtering, and as such will be used only as a thermal evaporator.

Anticipated problems:

1 – Blowing fuses – Ramp the current very slowly. Keep the current below 200 amps and the Fil Adjust below 50.

2 – Oil backstreaming – This would happen if the chilled water stopped flowing or if you open the hi-vac valve at the wrong time. Always touch the “out” line for the chilled water to make sure that it is still cool to the touch. Also make sure to open the valves in the appropriate order. **IF THIS HAPPENS THE CHAMBER WILL BE COATED IN OIL AND YOU WILL HAVE TO CLEAN IT!!!**

3 – Damage to bell jar – There is no winch on this system, and so users will need to lift it manually. Also, the bell jar can stick somewhat to the frame of the chamber. Be gentle with the jar, and make sure that when you lift it up you suspend it over the crystal monitor in case the bell jar slips.

4 – User error – If you aren’t paying attention, you will break something. Please don’t hesitate to ask for help. E-mail kyle.zufelt@gmail.com or call (801)422-6159.

Operating Procedure

1. Turn on the system power by pressing the black “On System” switch in the lower left hand corner of the control panel. Most of the buttons switch their function on and off (i.e. push once -> on, push again -> off), but the main power has a separate switch for on and off. The “Off Control” switch is immediately to the right of the “On System” switch.
2. Turn on mech pump power switch. Turn pressure gauge selector to "Foreline".

3. When foreline pressure drops below 50 mTorr, open backing valve. When pressure drops below 50 mTorr again turn on Hi-Vac Pump power switch (not the valve!). Wait 20 minutes for diffusion pump to stabilize. You may continue through step 6 while you wait.
4. Make sure that the rough valve is closed. Vent the chamber by turning on the nitrogen tank strapped to the wall and opening the vent valve. Make sure to leave the tank closed during and between runs as the line is leaky.
5. Raise the bell jar up to its highest point and rotate it to the right (so that it is suspended about an inch above the crystal monitor). Lower the bell jar about a centimeter until it supports itself.
6. If necessary, add more target material to the boat, placing something underneath the boat to catch any spilled material.
7. Put sample in chamber. Turn on the rotary power briefly to make sure that your sample is well attached and that there are no problems with the stage. Close bell jar.
8. Add LN2 through the funnel on the left of the machine until it starts to drip off the funnel (from frosted -> wet). Do not proceed to step 9 until the full 20 minutes have passed (see step 3).
9. Close backing valve, then open roughing valve. Start T_{rough} timer. Turn pressure gauge selector to "Chamber".
10. When chamber pressure drops below 100 mTorr, stop T_{rough} and record time on log sheet, close roughing valve, then open backing valve.
11. Open Hi-Vac valve. Start T_{HiVac} timer. Turn on high-vacuum gauge (not valve!). Record time at $5x10^{-5}$ Torr.
12. Wait until chamber pressure drops to $5x10^{-6}$ Torr.
13. Turn on rotary power and adjust speed to about 1/3 of the way open. You can see the rotary speed by watching the motor through the top of the bell jar.
14. In filament area of the control panel, the right switch should be set to the "right" boat and middle switch should be set to "Fil". "Fil Adjust" knob should be set to 0.
15. Turn on crystal monitor. Check to make sure the appropriate density and z-ratio have been input. (Iron – Density 7.86, Z-Ratio .349) Record XTAL% on log sheet.
16. Turn on "Fil/Glow Power" switch. Ramp up voltage (10%/30 sec) until desired deposition rate is achieved. Current should never exceed 200 A. “Fil Adjust” should not need to exceed 50.
17. Open shutter and zero thickness using controls on deposition monitor. When desired thickness is achieved, close shutter and turn down voltage. Turn off "Fil/Glow Power" switch, rotary speed and power, crystal monitor, and high vacuum gauge (not pump!).
18. **Close high vacuum valve!** Open N2 tank and then open vent valve.
19. Repeat steps 4-5 to open bell jar and remove sample. Turn off the N2 tank. Fill out an entry in the logsheet.
20. If you are going to deposit again, start from step 6.
21. If you are done for the day:
    a. Close the backing valve, open the roughing valve and rough the system down to 20 mTorr.
    b. Close all the valves, turn off the rough pump and the high vacuum pump, and turn off the power.

JIM

Thermal Evaporator
January 2012

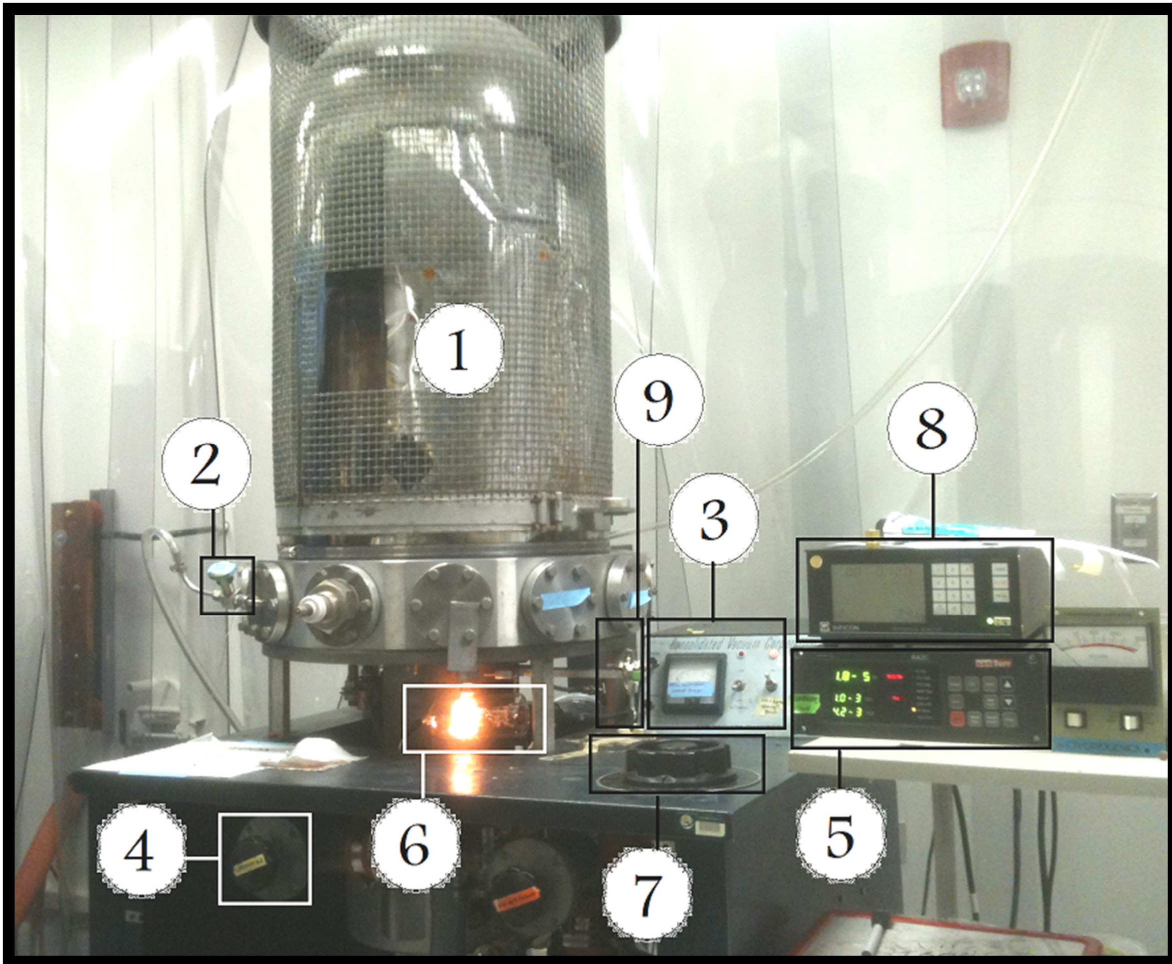


FIGURE A

FIGURE A-1 THRU A-9

1. **DEPOSITION CHAMBER**
2. **N_2 VENT VALVE**
3. **CONTROL PANNEL**
4. **ROUGHING PUMP VALVE**
5. **VARIAN SenTORR**
6. **ION GAUGE BULB**
7. **VARIAC**
8. **INFICON DEPOSITION MONITOR**
9. **SHUTTER LEVER**

FIG B. CONTROL PANEL –

Buttons on far left **RAISE** and **LOWER** Bell Jar

FILAMENT switch turns on the current, which is then controlled by the **VARIAC**

GATE VALVE switch opens and shuts the gate valve between the bell jar and the cryo pump as long as air is being supplied to the pneumatic lines

HOIST switch does not need to be touched. It should stay in the ON position

FIG C. The BOAT –

FIG D. CRYSTAL MONITOR

This Figure shows the crystal monitor and shutter inside the chamber.

----------WARNINGS---------

NEVER TOUCH THE FILAMENT OR FILAMENT PATH WHEN THE CURRENT IS ON

NEVER OPEN FORELINE VALVE

NEVER OPEN CRYOPUMP GATE VALVE WHEN THE CHAMBER IS NOT UNDER 1 TORR

NEVER FLOW MORE THAN 10 AMPS ACROSS THE FILAMENT

Current (VARIAC) Ramp rate should not exceed 20 V/min = 5V/15 sec. This is to prevent thermal shock to the boat, which will cause it to break.

N_2 TANK RELACEMENT = $14 + TIME

FILAMENT FUSE = TIME

BOAT = $50

I. GETTING STARTED

A. REVIEW THE WARNINGS SECTION IF YOU ARE NOT FAMILIAR WITH IT.

B. SYSTEM STANDBY - The Chamber should be left under vacuum when not in use. To begin using the chamber from an idle state, you will need to first vent the Chamber.

1. VENT THE CHAMBER AND RAISE THE BELL JAR

a. Ensure the Chamber is ready to be vented
   i. Make sure Ion Gauge in OFF (bulb is NOT lit). This is turned ON/OFF via the button labeled "Emis" on the VARIAN SenTorr. See FIG A-5.
   ii. Make sure Gate Valve is closed. The toggle switch on the Control Panel should be in the CLOSED position and the Gate Valve should have already been actuated to the CLOSED position since the last use of JIM. If the Gate Valve is closed, the chamber pressure should be greater than 1×10^{-3} Torr.
b. Open Nitrogen Tank located in the corner of the lab near the door. Be sure to CLOSE the Nitrogen supply when your work is complete. Record the starting Tank Pressure and ending Tank Pressure on the sheet just above the tank.
c. OPEN the Vent Valve. See FIG A-2
d. Press RAISE button to make sure the bell jar cords are tight.
e. When the rubber seal breaks (around 7.6×10^{2} Torr), the chamber has pressurized.
f. Press RAISE button to raise the bell jar.
g. Close Vent Valve.

2. PREPARE YOUR SAMPLE AND SOURCE MATERIAL

a. Tape your sample to the target plate with vacuum tape making sure it is where the Shutter will cover it.
b. If necessary, load evaporator boat between electrodes.
c. If necessary, add source material to the boat.
d. Make sure the Shutter is in the CLOSED position.
e. If needed, clean or change the glass slide window.

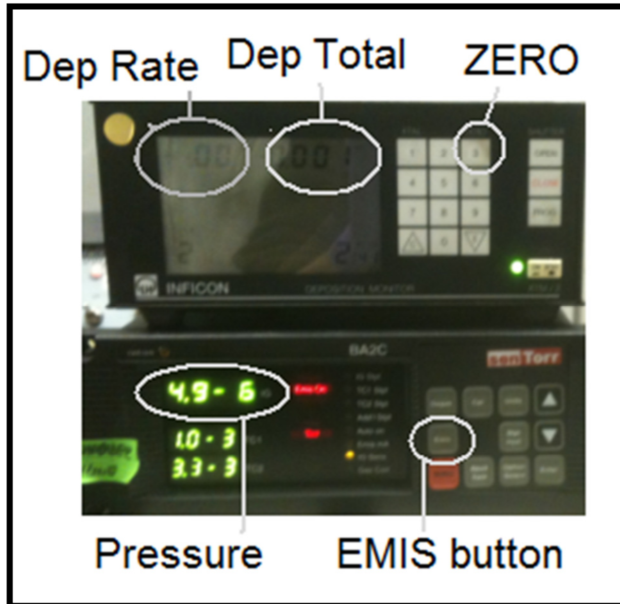
3. Check the Crystal Monitor (XTAL)

a. Turn on the monitor and check display for errors.
b. Make sure that the correct materials properties have been entered.
c. Push "XTAL" button (1). If XTAL reading is 100%, ask the student in charge of JIM to change the Crystal.

4. PUMP DOWN THE CHAMBER

a. LOWER the Bell Jar, making sure the jar is centered.
b. Make sure the Vent Valve is CLOSED.
c. Slowly OPEN the Roughing Valve until the Foreline Pressure reads 3×10^{2} Torr.
d. When chamber pressure reaches 3×10^{2} Torr, open the roughing valve all the way and start the T_{Rough} timer.
e. When the pressure reads 1.5×10^{0} Torr, CLOSE the Roughing Pump Valve.

f. RECORD the T_{Rough} on the time log.
g. OPEN the Gate Valve
    i. First, turn on the compressed air by opening the valve on the wall behind JIM. If the line is open you will hear a hissing noise and the regulator will read 75 psi.
    ii. Next, flip the Gate Valve Switch on the control panel up to the OPEN position.
    iii. Start $T_{cryo\ timer.}$ Turn off the compressed air.
h. Turn on the Ion Gauge by pressing the “Emis” button on the VARIAN SenTorr.
    i. The filament bulb should light up (FIG A-6)
    ii. The VARIAN SenTorr should now give a pressure reading next to the IG label. The “Emis On” should also be lit up.
    iii. Record T_{cryo} for the time it takes for the pressure to reach 5 x 10^{-5} Torr.
i. Allow Cryo Pump to reduce the pressure in the Chamber to 5 x 10^{-6} Torr.

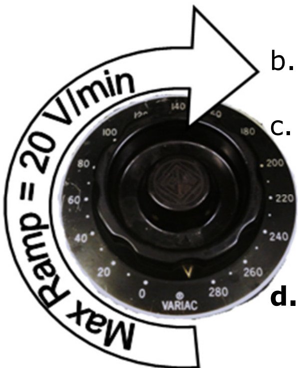


5. DEPOSITION

a. Make sure then INFICON Deposition Monitor is ON. (FIG A-8)
    i. Enter the material density and z-ratio

Material	Density	Z-ratio
Iron (Fe)	7.86	0.349
Aluminum (Al)	2.70	1.080
Lithium Fluoride (LiF)	2.60	0.779

    ii. Press PROG button when done to save changes.
    iii. Tooling Factor should be set at 109.5%.



b. Flip the toggle switch on the Control Panel labeled “FILAMENT” to turn on the current supply.
c. Ramp up the current supply at a rate of 20 V/min with the VARIAC (FIG A-7)
    i. The current supply should **NEVER** exceed 10 Amps. The current is monitored on the Control Panel.

d. Deposition Rate can be viewed on the Deposition Monitor (Figure A-8). Continue to increase the current until the deposition rate is at the desired rate.
e. **OPEN** the shutter manually while simultaneously PUSHING the **ZERO** button on the monitor. This will now record the thickness of the deposition on the sample.
f. **CLOSE** the shutter when the monitor reaches the desired thickness.

g. Ramp down the current supply with the VARIAC. Ramp down can occur at a faster rate than Ramp up but should not exceed 30V/min.
h. **RECORD** the Pressure, Voltage, and DepRate for the run in the logbook.

6. VENT THE CHAMBER AND REMOVE YOUR SAMPLE

a. Ensure the Chamber is ready to be vented
    i. CLOSE the Gate Valve. To do this, open the compressed air line, flip the Gate Valve switch to “closed”, and then close the compressed air line. You should hear a noise as the Gate Valve closes, and the pressure measured by the Ion Gauge should now be rising.
    ii. Turn off the Ion Gauge (bulb will turn off). This is turned ON/OFF via the button labeled “Emis” on the VARIAN SenTorr. See FIG A-5.
b. Open Nitrogen Tank located in the corner of the lab near the door. Be sure and CLOSE off the Nitrogen supply when your work is complete. Record the starting Tank Pressure and ending Tank Pressure on the sheet just above the tank.
c. OPEN the Vent Valve. See FIG A-2.
d. Press RAISE button to make sure the bell jar cords are taught.
e. When the rubber seal breaks (around 7.6 x 10^2 Torr), the chamber has pressurized.
f. Press RAISE button to raise the bell jar.
g. Close Vent Valve.

7. SYSTEM STANDBY

a. LOWER the bell jar, making sure it is centered to make a good seal.
b. Make sure the Vent Valve is CLOSED.
c. Slowly OPEN the Roughing Valve until foreline pressure reaches 3x10^2 Torr.
d. When chamber pressure reaches 3 x 10^2 Torr, open the roughing valve all the way and allow the chamber to pump down to 1 Torr.
e. CLOSE the Roughing Valve.
f. Write on the white board what material is in the boat.
g. REMEMBER TO TURN OFF THE NITROGEN BEFORE LEAVING THE LAB, and record the beginning and ending pressures in the N2 Tank.

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