Engineering Project Proposal

A Desktop Reactor for Plasma Enhanced Growth of Carbon Nanotubes

Team 23

Kyler Nicholson

John Taphouse

Janani Viswanathan

Bryan Yamasaki

Sponsors

Professor John Hart

Dr. Michael Fl De Volder

Eric Meshot

University of Michigan, Department of Mechanical Engineering

Section Instructor

Professor John Hart

December 9, 2008

1.0 Executive Summary

There are many potential applications for vertically aligned carbon nanotubes (CNTs), including various microelectronic and micromechanical devices. Vertically aligned CNTs, especially single isolated CNTs, cannot be consistently grown using pure thermal chemical vapor deposition (CVD) system. However, recent research suggests that the addition of plasma to CVD systems can greatly enhance the probability of growing vertical CNTs. Working with Professor John Hart, Dr. Michael Fl De Volder, and Eric Meshot of the Mechanosynthesis Laboratory, our team is to design and build a desktop sized plasma enhanced chemical vapor deposition (PECVD) system.

To achieve this goal our team systematically identified customer requirements and quantitative engineering specifications. The customer requirements and engineering specifications were analyzed using a quality function deployment (QFD) diagram, which identified three key customer requirements:

- *Control of Operating Conditions*: provides control over temperature, pressure, plasma, and flow rate
- *Adjustable Electrode Gap*: provides variability in electric field conditions
- *System Size*: system should be able to fit on a desktop

Background research and system level benchmarking revealed that PECVD systems can generally be broken down into 3 modules and one submodule of design: reaction chamber, plasma coil and electronics, operating condition controllers, and the substrate holding and heating assembly respectively. The customer requirements and specifications were used to guide the formation of preliminary design concepts for each module.

The final system design fits on an 18 by 18 inch base plate and is no more than 12 inches tall. The reaction chamber is composed of three mutually orthogonal tubes intersecting at their midpoint. Attached to the end of one of the tubes is a quartz tube. The plasma coil will be wrapped around the quartz tube and ignite the reactant gases before entering the reaction chamber. The entire substrate holding and heating mechanisms and the adjustable electrode are packaged onto a single tray that can be easily slide in and out of the reaction chamber. The chamber and infrared sensor are conveniently packaged on a stand with a linear bearing for opening and closing the chamber. A complete schematic can be seen in Figure 16.

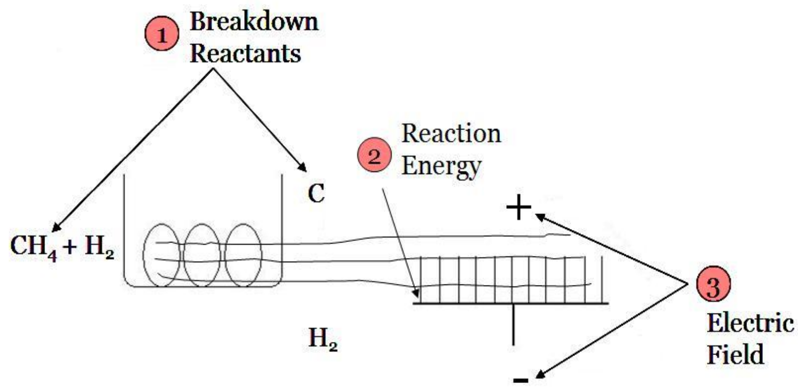
The entire chamber will be composed of prefabricated commercially available components. The only system components that will require machining are the system tray, electrode, substrate holding heat sinks, and system stand. The system tray and substrate holding heat sinks will be machined professionally due to their intricate geometry. The electrode and system stand will machined by the project team using waterjet cutting techniques. Once, all manufacturing is complete the project team will assemble the entire system by hand.

2.0 Table of Contents

3.0 Background

The University of Michigan's Mechanosynthesis laboratory under the direction of Professor John Hart and Dr. Michael Fl De Volder, is dedicated to the development of processes for fabricating carbon nanotubes (CNTs). CNTs show immense prospect in applications across many industries, including microelectronics and advanced composites. For many of these applications the growth of isolated vertical CNTs is required. Chemical vapor deposition (CVD) processes are currently commonplace for growing CNTs. CVD is capable of growing dense forests of tall CNTs, like those seen in Appendix A., but are unable to produce isolated vertical CNTs. Recent research suggests that the addition of plasma to the CVD process can greatly enhance the probability of growing vertical CNTs. The addition of plasma provides three main benefits as shown in Fig 1 below. It aids in the decomposition of the reactant gases for producing the nanotubes, creates an electric field that aligns the CNTs vertically, and adds energy to the reaction.

Fig. 1: Three benefits of plasma to a chemical vapor deposition system



Due to the Mechanosynthesis group's lack of experience in plasma generation and processing we were asked to develop a prototype plasma-enhanced chemical vapor deposition (PECVD) system. Upon completion of the prototype, we have also been asked to confirm its ability to fabricate vertically aligned carbon nanotubes and to display electron microscope images of the CNTs grown using this technique at the design expo.

Background research was conducted and it revealed that there are four primary methods for generating plasma in PECVD systems: Direct current (DC), RF triode, inductively coupled, and microwave. The benchmarked designs for each of these methods can be seen in Figs. 2-5 below.

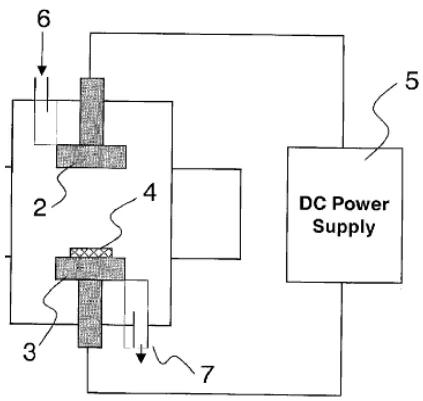


Figure 2: DC Powered PECVD

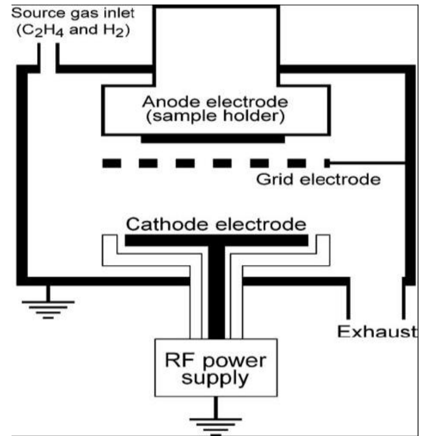


Figure 3: RF Triode Powered PECVD

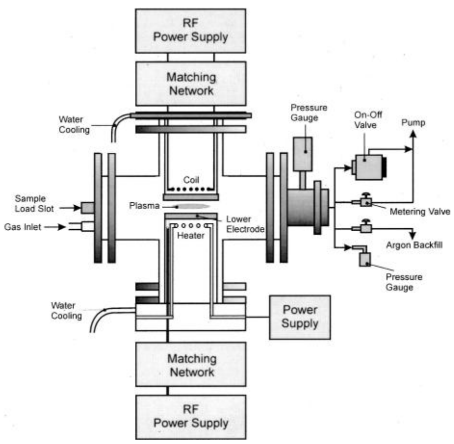


Figure 4: Inductively Coupled PECVD

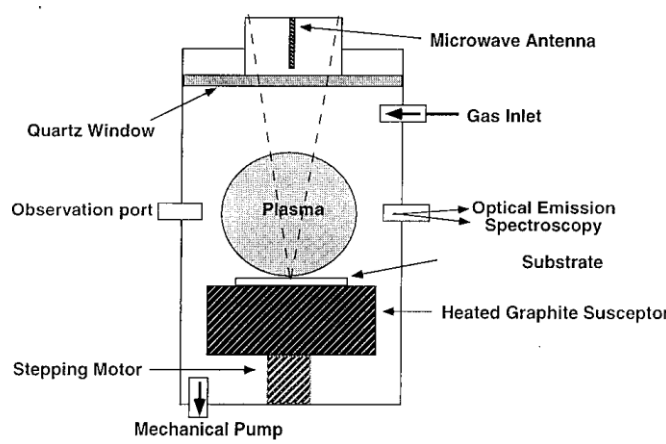


Figure 5: Microwave Powered PECVD

A comparison of the operating parameters for each method can be seen in Appendix C. The research also showed results for improved vertical alignment for all three of the plasma generation methods. Finally, two strategies were identified for the PECVD design based on the background research and benchmarked designs: local and remote. Details for this are covered in section 5.2 of the report. All of the information sources used for the background research are listed in references.

4.0 Customer Requirements and Engineering Specifications

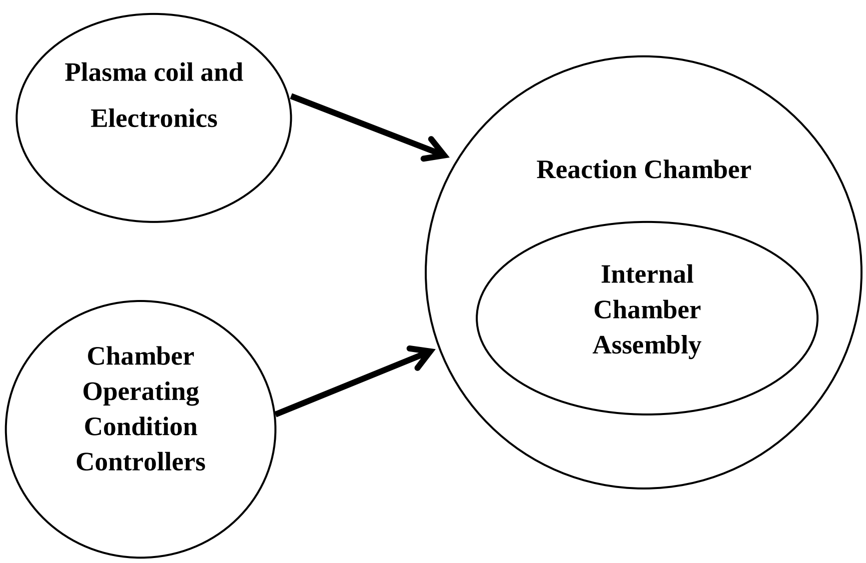
The design of the system was guided by customer requirements and engineering specifications. The customer requirements were gathered through meetings with the sponsors and can be seen in Appendix B. To better guide the design the relative importance of each customer requirement was determined through a pairwise comparison, as seen in Appendix D. The customer requirements were then translated to quantitative engineering specifications through benchmarking of existing PECVD designs and consultation with the project sponsors. Additionally, the customer requirements and engineering specifications were analyzed using a quality function deployment (QFD), Appendix E. The QFD identified key aspects of the design, as well as the strength of correlation between each requirement and specification. The most important design aspects as identified by the QFD are:

- *Control of Operating conditions*: provides control over temperature, pressure, plasma, and flow rate
- *Adjustable Electrode Gap*: provides variability in electric field conditions
- *System Size*: system should be able to fit on a desktop

5.0 Modules and Strategies of Design

Background research and benchmarking revealed that PECVD systems can be broken down into three basic modules of design and one submodule: the reaction chamber, the plasma coil and supporting electronics, the chamber operating condition controllers, and the internal chamber assembly respectively. The reaction chamber is the central module, within which is the sub module internal chamber assembly. The plasma electronics and operating condition controllers are external modules that support the function of reaction chamber module, as seen in Figure 6 below. Our research also revealed that PECVD systems are generally designed along either remote or local plasma strategies.

Figure 6: The reaction chamber is the central design module, within which is the internal chamber assembly submodule. The plasma coil and electronics and operating condition controllers support the function of the reaction chamber.



5.1 Design Modules

The reaction chamber is the central component of all PECVD systems and one of the most effort intensive design modules. It is the chamber in which the plasma is generated and the entire nanotube growth process takes place. It includes the chamber walls, viewports, reactant gas inlet and outlet, as well as electrical feed through. All of the remaining design modules and submodule are largely dependent on the design of the reaction chamber. The plasma coil and supporting electronics and the operating condition controllers are particular to the method of plasma generation, which is the dominant influence in the design of the reaction chamber. Additionally, the internal chamber assembly submodule is largely dependent on the interior geometry of the reaction chamber. For these reasons concepts were first generated for the reaction chamber.

After a concept was selected for the reaction chamber design, development efforts were focused on the design of the remaining modules and submodule. The design of the plasma coil and supporting electronics involves design of the coil and selection of a power supply and matching network. The design of the operating condition controllers involves selection of a pumping system, fittings, and an infrared sensor. The design of both of these modules is largely inherent to the chosen plasma generation method and reaction chamber design. The internal chamber assembly submodule entails the design of the entire interior of the chamber, including, the electrode, substrate holding mechanism, and resistive heating element. For this submodule a preliminary concept was generated, as seen in Appendix F, and then iterated until a final design was reached. During the design of the internal chamber assembly the electrode assembly was identified as a design submodule required additional design work.

5.2 Strategies

Based on background research and benchmarked designs we identified two sensible strategies for PECVD design: local and remote. In local plasma PECVD systems the plasma is produced directly over the substrate in the growing environment. On the other hand, in remote plasma PECVD systems the plasma is formed away from of the growing environment and then channeled to the substrate. Each strategy has its associated advantages and disadvantages shown in Table 1 below.

Table 1: Side by side comparison of remote and local plasma strategies

	Remote Plasma	Local Plasma
Advantages	No sputtering over substrate. Separate control of plasma and electric field.	Electric field over substrate.
Disadvantages	Weak electric field over substrate.	Sputtering can damage substrate or nanotubes.

The main disadvantage of the remote plasma strategy, a weak electric field for vertical alignment of the nanotubes can be surmounted by introducing an independently created electric field around the substrate. For this reason, the majority of our preliminary design concepts use the strategy of remote plasma generation.

6.0 Brainstorming and Concept Generation

To generate our preliminary concepts each team member independently sketched two designs. The designs were then reviewed and a discussion took place to improve and build upon the designs. The revised designs were then presented to Dr. Michael De Volder who evaluated them. Dr. De Volder helped to revise the existing concepts and through discussions several entirely new concepts were developed. In total seven concepts were created and can be seen in section 7.

7.0 Concept Summary

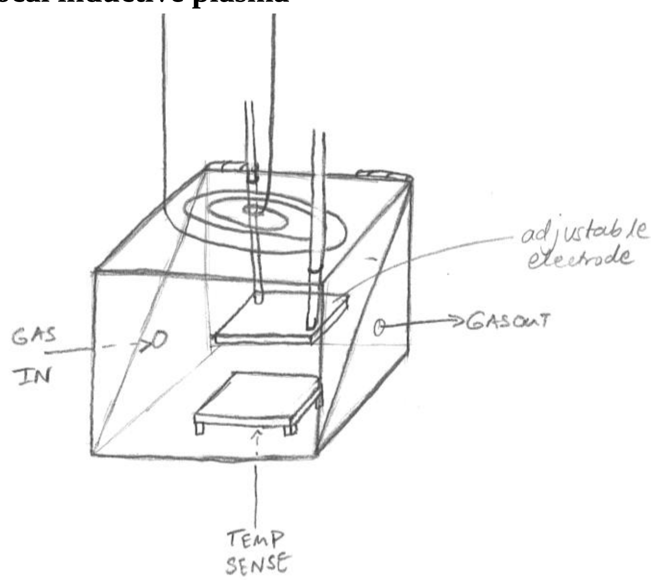
In total, seven concepts were generated and can be seen below in Figures 7-13. Two concepts were generated for each type of chamber geometry: a box, tube or cross and one “wild idea”. The concepts utilized both local and remote plasma strategies. Chamber materials were also given some consideration, as either stainless steel or quartz.

One key module of our design is the adjustable electrode. Two concepts for achieving an adjustable electrode are shown in section 7.2.

7.1 System Concepts

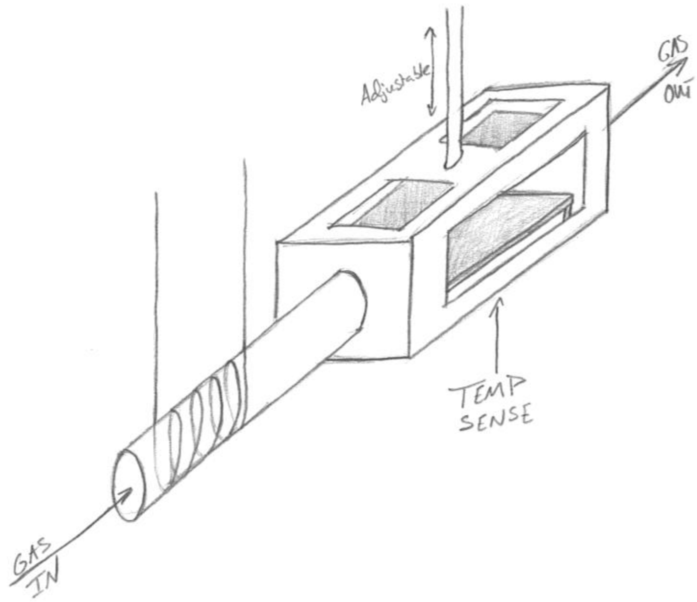
Shown below are the seven concepts we developed for the system.

Figure 7: Box with local inductive plasma



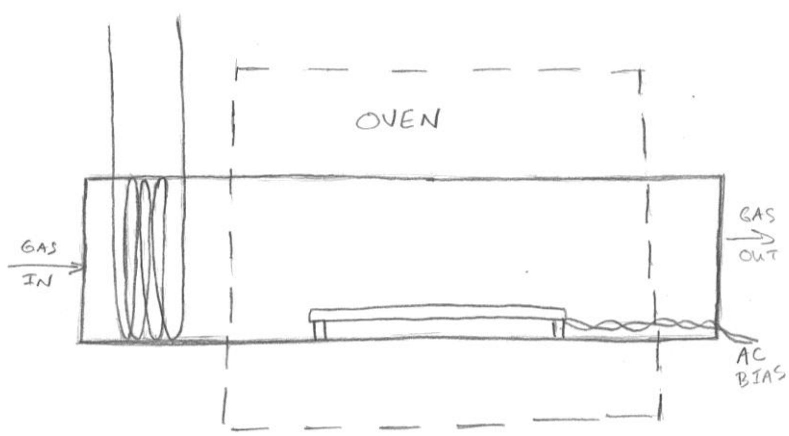
The box with local plasma system uses an antenna wrapped in the shape of a stove heating coil. This coil is placed outside the chamber on a quartz window to excite the plasma gas locally. The front is opened like a clam shell to increase the access to the substrate.

Figure 8: Box with remote inductive plasma and DC substrate bias



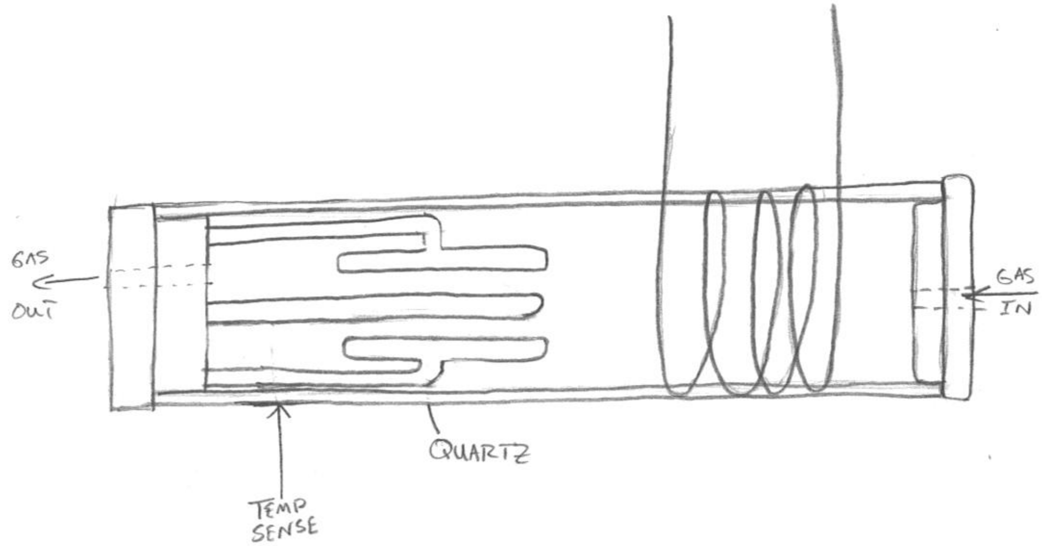
The box with remote inductive plasma creates remote plasma in the tube which moves through the chamber. The remote plasma allows for disjoining the plasma source and electric field. The user would open the end of the box where the gas outlet is to access the substrate.

Figure 9: Tube with remote inductive plasma and oven heating



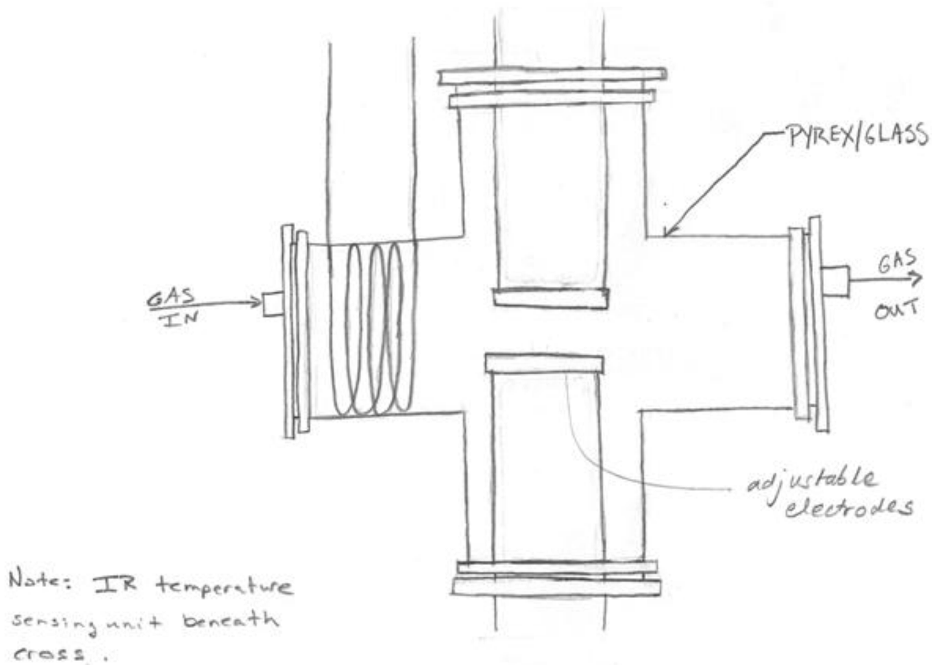
Tube with remote plasma adds plasma to a CVD system, where an oven is used as the heating source. This setup is currently used in the lab. A stand for the substrate which is electrically biased and a coil wrapped around the inlet of the tube would be the major additions to the current setup.

Figure 10: Tube with remote inductive plasma and DC substrate bias



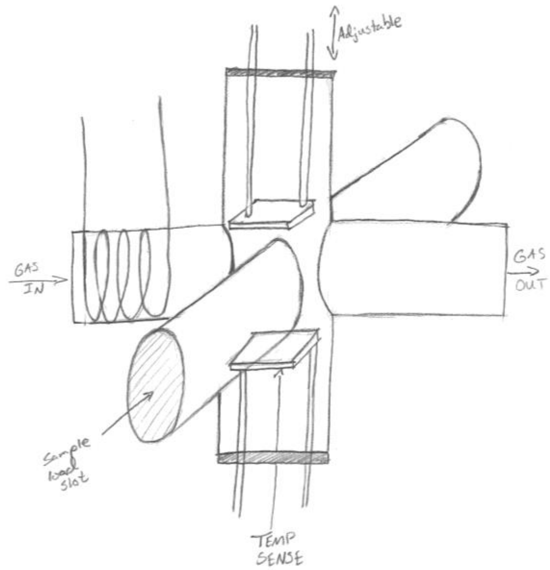
This tube design uses a single quartz tube, where plasma is generated in one end using a coil. The electrodes are attached to a cap on the opposite end where a voltage can be applied. This is a modification of a design currently used in the Mechanosynthesis Lab.

Figure 11: Pyrex cross with remote inductive plasma and DC substrate bias



The Pyrex cross design is made from commercially available Pyrex cross. One arm is used to generate the plasma, while the other for loading the sample. This design has been used in many research labs.

Figure 12: Pyrex or steel six-way cross with remote inductive plasma and DC substrate bias



The six-way cross design can be made in a couple different ways. Three holes are machined in a block of stainless steel, or three Pyrex tubes are attached in a cross fashion, or a stainless steel chamber can be purchased from a vacuum system supplier. The extra arms can be used for viewports, as well as having more adjustability later for different configurations.

Figure 13: Spherical Pyrex and steel chamber with local plasma source

The sphere is our “wild idea,” which uses a hemisphere of Pyrex to allow the viewing of the substrate during nanotube growth.

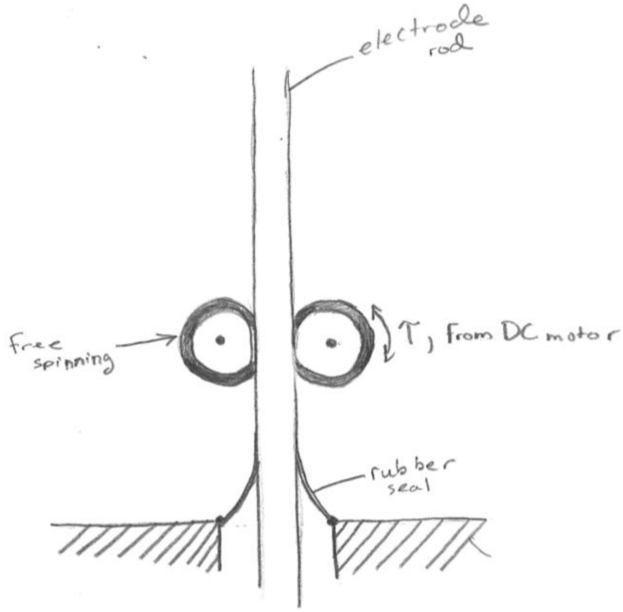
7.2 Key Submodules

Preliminary concepts were also generated for two key submodules: adjustable electrode and internal chamber assembly.

7.2.1 Adjustable Electrode

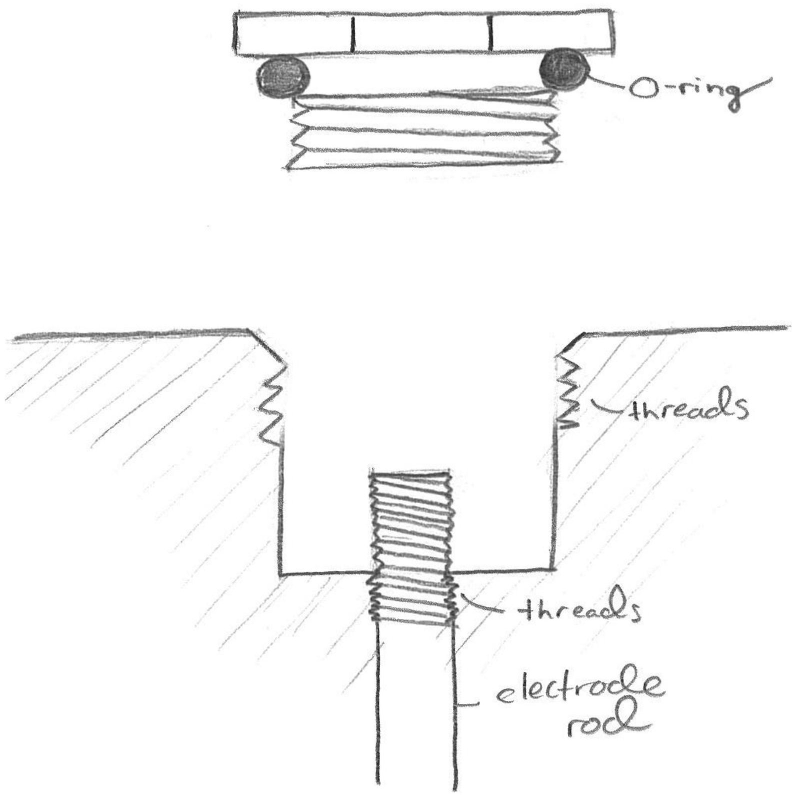
One key module that was determined for the system is the adjustable electrode. Below, two of our preliminary concepts are shown to achieve an adjustable electrode.

Figure 14: Automatic Height Adjustment Concept



The automatic height adjustment concept uses a DC motor to rotate a wheel that raises or lowers the electrode. A difficulty of this design is that the seal needs to be dynamic to allow the rod to move and still keep the pressure difference between the chamber and atmosphere.

Figure 15: Manual Height Adjustment Concept



The manual height adjustment uses a threaded rod on which the electrode is attached and by rotating the rod the electrode will move up or down. A cap with an o-ring is secured over the rod which seals off the chamber from atmosphere.

7.2.2 Internal Chamber Assembly

The internal chamber assembly was identified as a key submodule of design. A preliminary concept was generated for this module and is shown in Appendix F. The concept was then reviewed and refined through discussion with the sponsors until a final design was achieved.

8.0 Concept Screening and Selection

This section will go into detail on how we developed and utilized a screening process to determine our top two system concepts. We observed how current systems are used in the lab as well as talked to current users of CVD systems, to help us pick a winning concept. We have also made refinements to this concept since DR2 to make it a more practical design.

8.1 Concept Screening

To evaluate our seven system design concepts, we decided to rate them against five of our critical criteria to determine the top two choices. The five criteria we used are: Cost, Ease of Fabrication, Adjustability, Size and Access to Substrate. The cost is a concern, to keep the project budget at a reasonable level for a one-off PECVD system. The ease of fabrication is important, because we need to be able to make and assemble the system, and if that is not possible, then the customer requirements cannot be met. The adjustability of the system helps to increase the number of ways the system can be used, thus decreasing the cost of ownership to the customer. The size is important, because the desktop size is a major customer requirement. The access to the substrate is needed, to let the user easily swap out samples and to reduce time between experiments.

Each design concept was rated a zero, one or two for each criteria; zero meaning bad, one is fair and two is good. This methodical process helped us think deeply about each design and determine the strengths and weaknesses. After rating each concept and totaling up the score the top two design concepts were: Box with remote plasma and steel six-way cross. Below, Table 2 shows the complete comparisons of our design concepts.

Table 2: Box with remote plasma and the steel tri-cross are the leading design candidates

	Box (Local plasma)	Box (Remote plasma)	Tube (Oven heating)	Tube (Local heating)	Cross (Pyrex)	6-way Cross (steel)	Sphere
Cost	2	2	2	2	1	2	0
Ease of Fabrication	1	1	1	2	1	1	0
Adjustability	0	2	0	0	1	2	0
Size	1	1	1	2	1	1	2
Access to Substrate	2	2	1	0	0	1	2
0 – Bad, 1 – Fair, 2 - Good	**6**	**8**	**5**	**6**	**4**	**7**	**4**

8.2 Observations and Discussions

To help decide between the two leading design concepts, we discussed with lab users Dr. De Volder and Eric Meshot. They both have used a variety of CVD setups and know what features are helpful to increase the usability of a system. We also watched Dr. de Volder setup and begin growing CNTs, which helped us see how users handle samples.

To help decide between local and remote plasma we met with Professors Foster and Kushner, who both do research involving plasmas. They helped us establish the benefits of local and remote plasma sources and provided consultation for choosing a plasma generation method.

8.3 Final Concept

Consultations with Professor Foster and Professor Kushner were instrumental in deciding to use a remote inductively coupled plasma source. Having a remote plasma source will allow decoupling of the plasma and electric field control, providing increased adjustability to the system. The inductively coupled plasma source comes at a lower cost than microwave plasma and operates more cleanly than a DC plasma source.

Throughout our observations and discussions with Dr. De Volder and Eric Meshot, we determined that any benefit of having a box shape, which would have a better access to the substrate, were out weighted by the difficulty we would have to close up and seal the chamber for vacuum. We also determined that having six arms in a cross would allow for greater adjustment in the future. For these reasons we chose to go forward with the six-way cross.

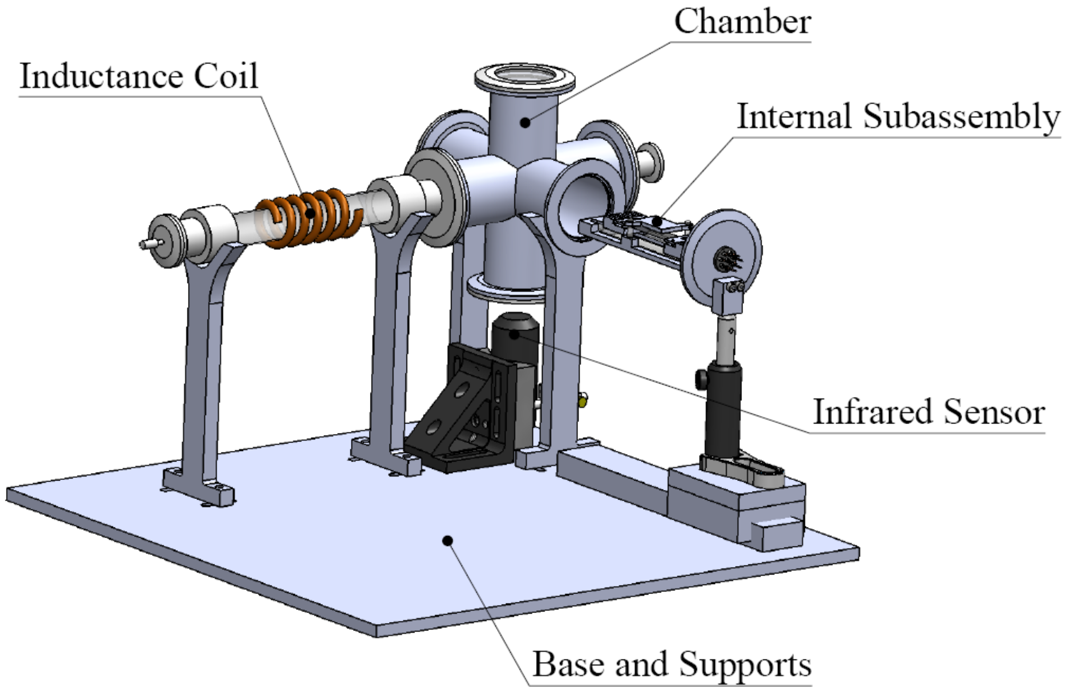
Due to the short time frame of this project, we decided to purchase parts for the chamber from vacuum component companies. This ensures that all of our fittings will hold vacuum, will be easy to assemble and will decrease the amount of time we need to spend machining parts.

The decision to use inductively coupled plasma required that a quartz tube be attached onto the end of the gas inlet arm of the cross for plasma generation. The conductive properties of stainless steel would create an issue if we were to wrap a coil around it and try to ignite plasma. The use of a quartz tube to generate plasma in is also beneficial, so that the plasma can be seen and monitored easily.

9.0 Final Design

The complete system can be seen below in Figure 16. We broke up the final design into three main subassemblies. The first subassembly is the reaction chamber (Figure 17). This includes the reaction chamber body and the quartz tube in which the plasma is generated. The second is the internal chamber assembly (Figure 18). This includes all the substrate holding and heating mechanisms and the electrode. The final subassembly is that of the base and supports (Figure 20). This includes all chamber supports, IR sensor and support, internal system tray support, and the base to which all supports will be attached.

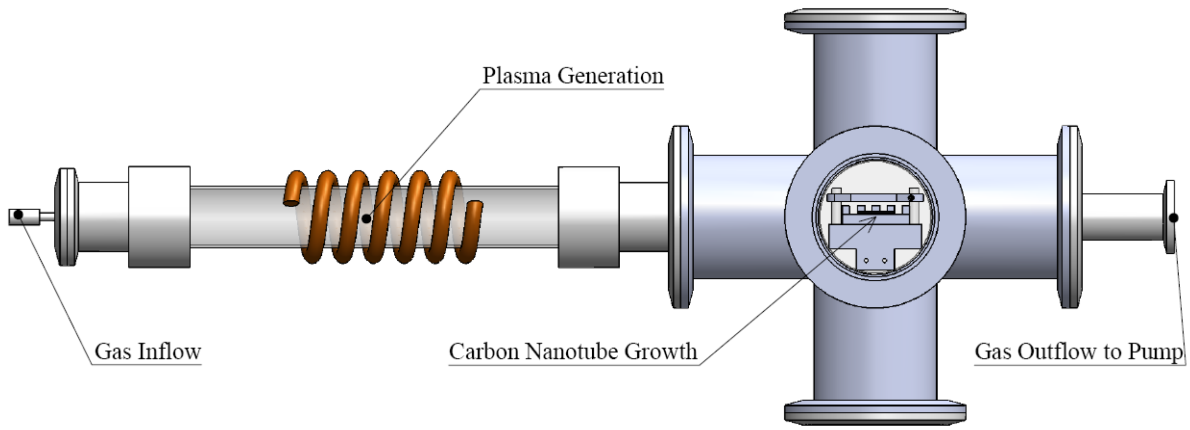
Figure 16: Final system design, composed of three subassemblies



9.1Chamber Design

For the design of the chamber, we chose a 2 inch diameter six-way cross geometry (purchased from Lesker), due to its versatility and wide selection of standards components. All flanges meet the standards for Klein Flanges (KF) by the International Standards Organization and can therefore be connected together with ease. The gas flow will enter through a Swagelok fitting as seen on the left side of Figure 17. The flow of gas will then travel through a 1 inch diameter quartz tube. This tube will be attached to the system with two quick connect fittings, facilitating the ease of interchangeability to new clean tubes and tubes of different length. A copper wire will be wrapped around this tube and connected to the RF generator for creating the plasma. A second quick connect will be used for attaching the quartz tube to the left side of the six-way chamber. The top and back flange of the chamber will have kodial glass viewports for viewing of internal components, while the bottom flange will have a kodial glass viewport for optical access for an IR sensor. The front flange will be for the electrical feedthrough, which consists of eight individual pressure sealed copper pins. The gas will then flow out through a standard KF flange connected to a steel hose, which is in turn connected to a vacuum pump.

Figure 17: Chamber design subassembly



9.2 Internal Chamber Assembly

The internal chamber assembly, seen below in Figure 18, is for providing support to all the components used for the growth of the carbon nanotubes. The backbone of this subassembly is the system tray, which is fixed to the electrical feedthrough flange using steel dowel pins and set screw collars. The dowel pins with be press fit through the flange and slip fit through the system tray. The bottom three electrical feedthrough pins will be clipped off to avoid interference with the system tray.

Sitting inside two grooves in the system tray are two quartz rods. The function of the rods is to support the substrate assembly and to keep it electrically isolated from the system tray and chamber. The substrate assembly consists of two heat sinks and a silicone resistive heating element, which will support the substrate. The function of the heat sinks is to hold the silicone heating element and to pass current through it, which will in turn heat up the substrate. A closer look at the heat sink construction can be seen in Figure 14 below. The silicone heating element sits directly on the heat sink; a small blank plate sits on top of that. The top plate has two through holes and one threaded hole. The two through holes are to screw the top plate to the heat sink while the middle threaded hole is to screw a thumb screw into the middle plate. The added pressure of the thumb screw on the middle plate ensures a large contact area of even pressure on the silicone heating element.

An electrode will be used to create an electric field and will be supported by two quartz rods set into the system tray vertically. The height will be controlled by a set of silver tipped set screws. One of the electrical feed through pins will be connected to this electrode. Each heat sink will also be connected to feed through pins as well. The last two pins are to be used for a removable thermocouple to calibrate the IR sensor. The actual connection wires and clamps are to be determined.

Figure 18: Internal Subassembly

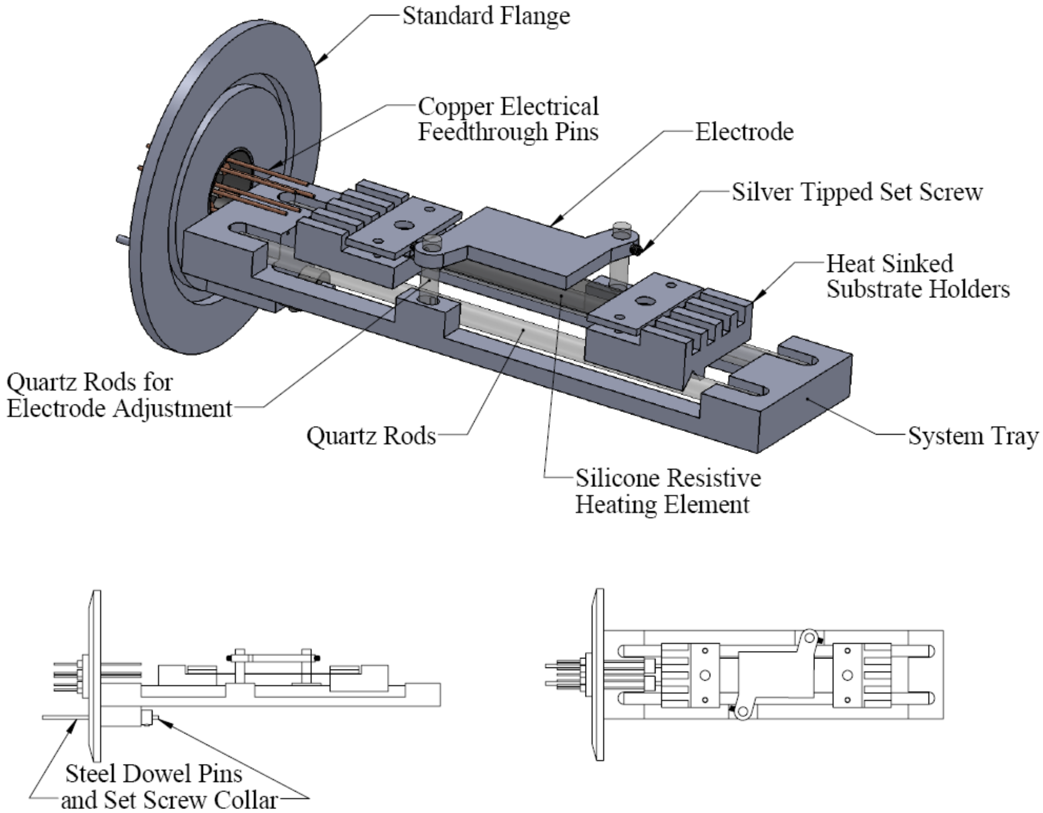
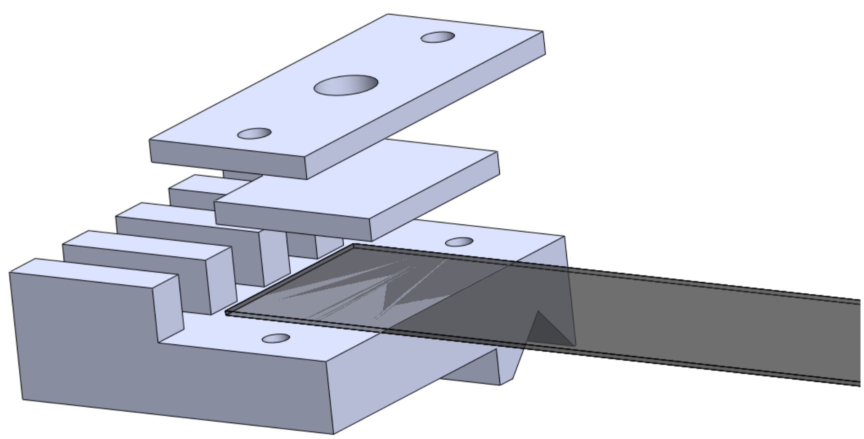


Figure 19: Exploded view of heat sink construction



9.3 Base and Supports

The purpose of this subassembly is to support the chamber, the IR sensor, and the system tray. The base plate will composed of a flat piece of aluminum. The chamber supports will be mounted to this plate. These supports will keep the chamber at a fixed height. The IR sensor stand will also be screwed to the base plate. The IR sensor stand allows for vertical adjustment of the sensor to properly calibrate it to the appropriate height. The system tray holder will keep the internal subassembly with the system tray at a controlled height. This holder allows for vertical motion to adjust the stand to align properly with the chamber. The holder will also slide on a rail to be able to pull the system tray completely out to of the chamber, as seen in Figure 20 below. This is beneficial for loading and unloading substrates for the growth of carbon nanotubes, replacing silicon heating elements, and removing of entire system tray for cleaning. The holder will be connected to the internal subassembly through a plate that clamps onto the push fit dowels in the electrical feedthrough flange as mentioned in the previous subsection.

Figure 20: Base and supports

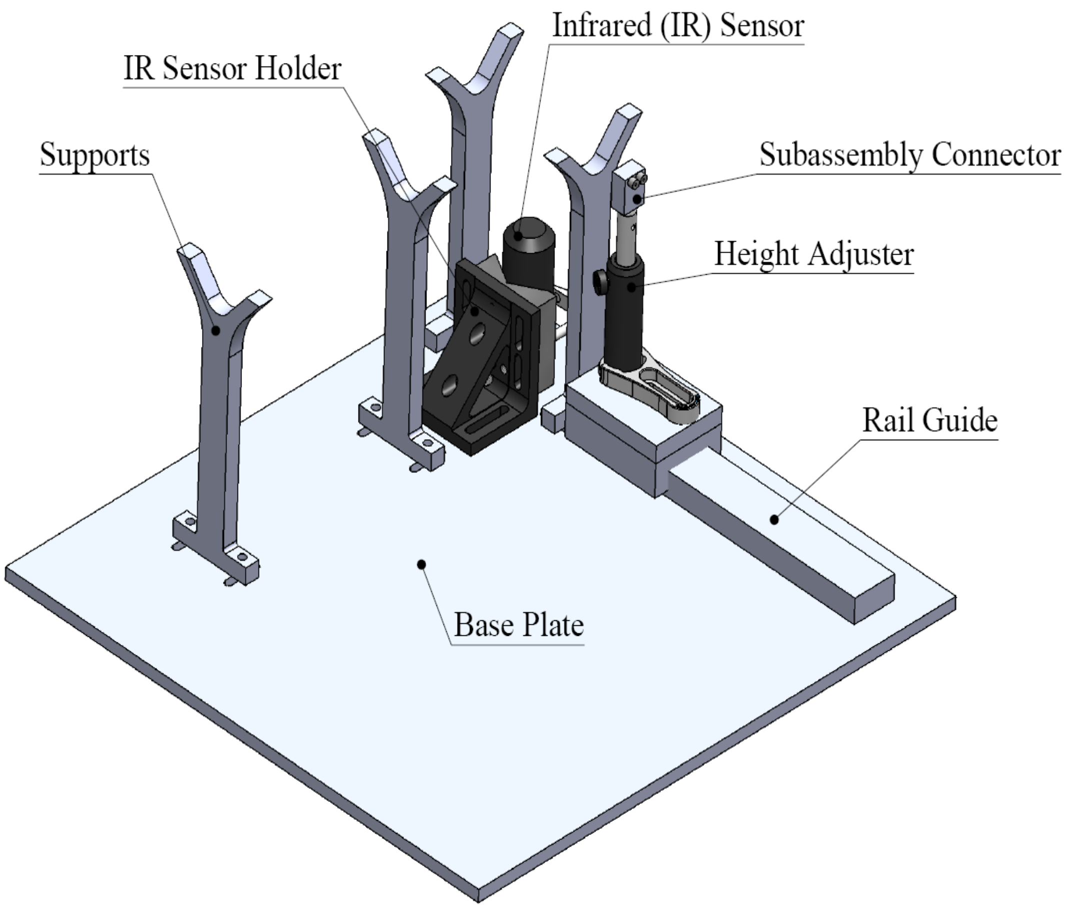
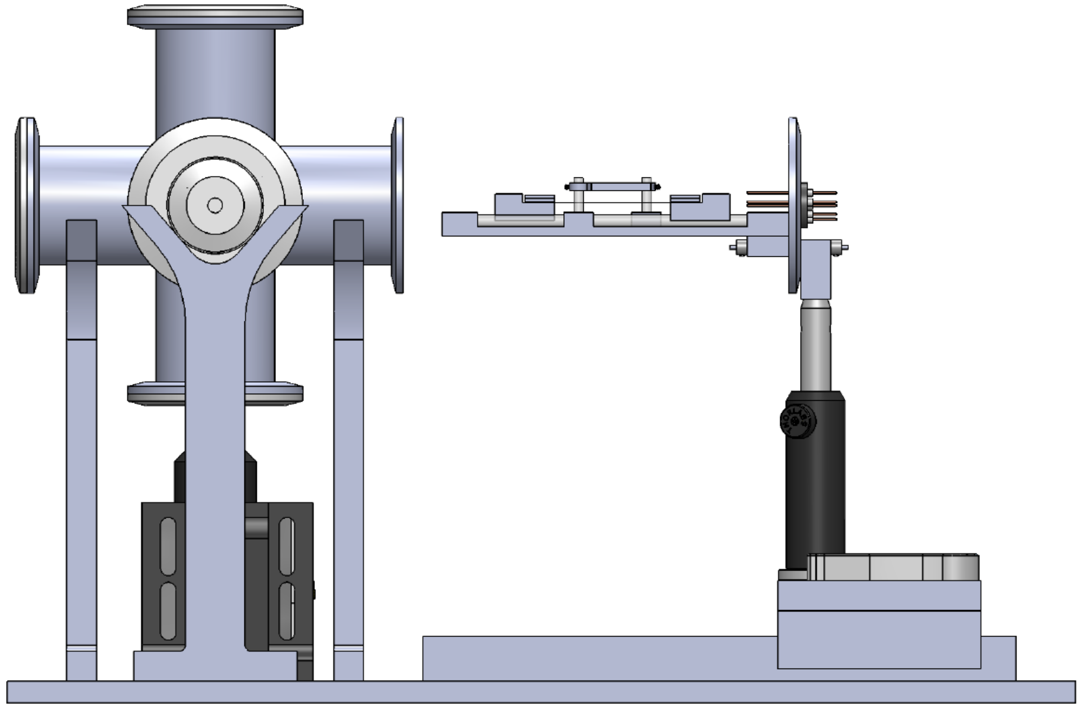


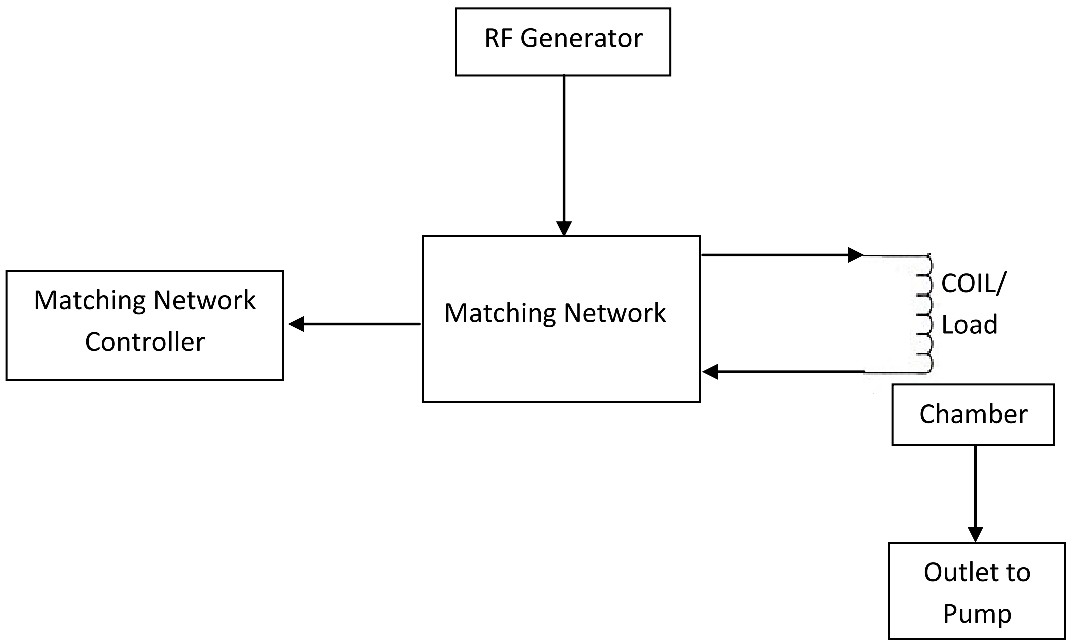
Figure 21: View of internal subassembly completely removed from chamber by sliding on rail



9.4 Electronic Design and Set-up

Fig. 22 below shows the electrical connections for the reactor system. The inductive coil wrapped around the quartz tube is connected in parallel to the shunt capacitor in the matching network. The matching network is in turn connected to the RF power generator and an automatic matching network controller. The automatic matching network tunes/ matches at any given time the impedance of the load (plasma) to that of the source (generator). It has to be ensured that the coil and the matching network have adequate housing with ground connections in them, to reduce the risk of being electrocuted. The entire setup of the tube and the coil is wrapped around by a copper mesh which acts as a faraday's cage to reduce the interference of the RF from any other surrounding equipment and the generator itself.

Figure 22: Representation of the Electrical set-up for the reactor system



9.5 Bill of Material

Appendix G provides a summarized table listing the part numbers and descriptions, vendors, list prices and lead times for all system components. Of our parts, the weldable flange and feedthrough is the only custom part and has a reasonably short lead time of one week. One of the most expensive parts is the RF generator and the matching network, costing $5000. The RF generator and the matching network is a refurbished model capable of 500W. Its price is competitive with the price of a new generator rated for 300W without a matching network. While only 300W was determined necessary from the parameter analysis, 500W gives us flexibility in operating the generator over a wide range of power. The matching network is necessary for the system because it transforms the steady state impedance of the load (plasma) back to an impedance which is optimum for the RF generator (50 Ω) and protects (buffers) the RF generator from unstable load conditions. If the load is not perfectly matched, then some of the power is reflected back to the generator. The most expensive part of the system is the vacuum pumps, costing approximately $7,000. In order to meet the customer specifications for flow rates and the low vacuum pressure, two pumps were required. However, for the purposes of our project, we decided to use the pump provided to us by our sponsors. This will run at a pressure of 0.6 torr, instead of the 0.1 torr that new pumps would have provided. This will affect the plasma

generation process and the growth process in turn, but for the purposes of our project this was deemed as an effective solution for the testing stage and considerably lowers the cost.
Finally, 6061 aluminum was chosen to manufacture the base plate and the support for the chamber. Two aluminum plates measuring 18" by 18" will be required to machine these parts.

9.6 Engineering Design Parameter Analysis

Plasma modeling was carried out, with the help of Professor Mark Kushner in the EECS department, to determine the effect of input parameters (inputs) such as power and pressure. Based on the results, the power to be supplied, the current through the inductive coil and the chamber pressure was determined. The modeling also provided a check to ensure that the maximum temperatures reached by the quartz tube and the chamber did not exceed the maximum service temperatures of the materials. By inputting different dimensions of the chamber and the quartz tube, the modeling enabled us to view the effect of changing geometries, and thus allowed us to choose dimensions which produced reasonable results. Additionally, calculations using the through-put formula were performed with the specified flow rate and pressure. This enabled us to determine the pump type and specifications such as the pump speed. The sections below describe in further details the analysis carried out for the power selection, the pump capacity, and the chamber dimensions.

9.6.1 Parameter Analysis Examples

Power selection

The modeling was carried out for argon gas for two primary test cases: 50 W and 300 W at a pressure of 0.1 torr and 100 sccm. The electron density, the temperature of the reactant gas (argon) and the total power deposited to all the excited states of the gas and the electrons (load) was considered at these powers. The figures below show that the electron density, the temperature, and the total power deposited to the load were higher for the 300W simulation than the 50 W simulation. At 300 W, the temperature of the argon gas in its neutral and excited states averaged at 70°C, which was well within the maximum service temperature range of 1100 – 1400°C for the quartz tube and 250 – 300°C for the O-ring connecting the tube to the chamber. The temperature of the neutral gas species was calculated by imposing a constant temperature boundary condition of 52°C on the quartz tube wall. Once the temperature of the neutral gas was determined, it can then be safely assumed as the wall temperature in the absence of any active insulation i.e. a temperature gradient now exists between the wall and air, instead of the neutral gas and wall (Refer to Appendix H for temperature plots for argon in its neutral and excited states). Moreover, it can be seen from Figure 23a that the electron density extends into most of the inner chamber without the aid of the gas flow due to the pump and thus the substrate is continually exposed to the plasma during the process. We also noticed from the modeling that the total power deposited to the load was approximately 80 W/cm^3. This means that over the 2 cm length of the tube shown in Fig. 23b, the total power deposited to the plasma load can be determined as,

$$P_{TOT} = \int P\left(\frac{W}{cm^3}\right)\cdot dVolume = \frac{1}{2}P\cdot\pi r^2 h \approx 240W \qquad \text{(Eq. 1)}$$

This is close to the source power of 300 W and provides a check for the above results, validating the accuracy for the set of conditions imposed (pressure, temperature, chamber and coil dimensions and flow rates). This consequently also enabled us to determine the maximum current that could flow through the inductive coil to be approximately 10 A.

Figure 23: Contours plots depicting a) electron density and b) total power deposited to plasma at 300W

a. b.

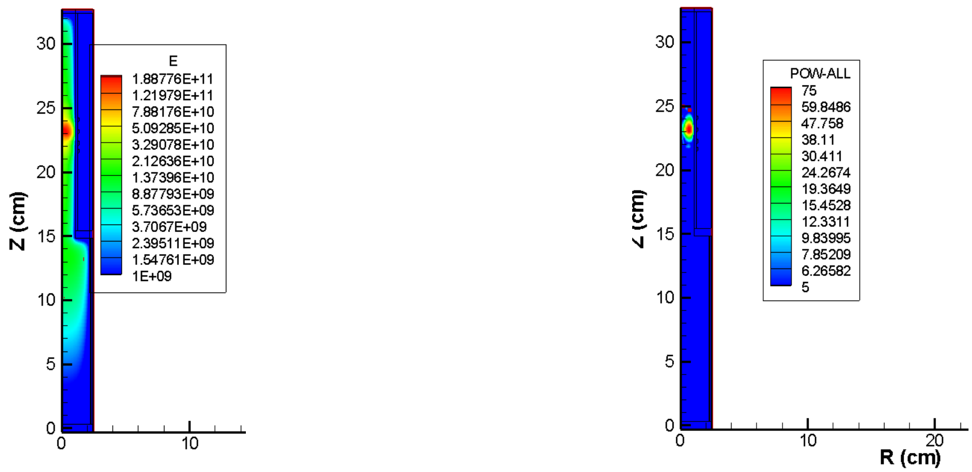
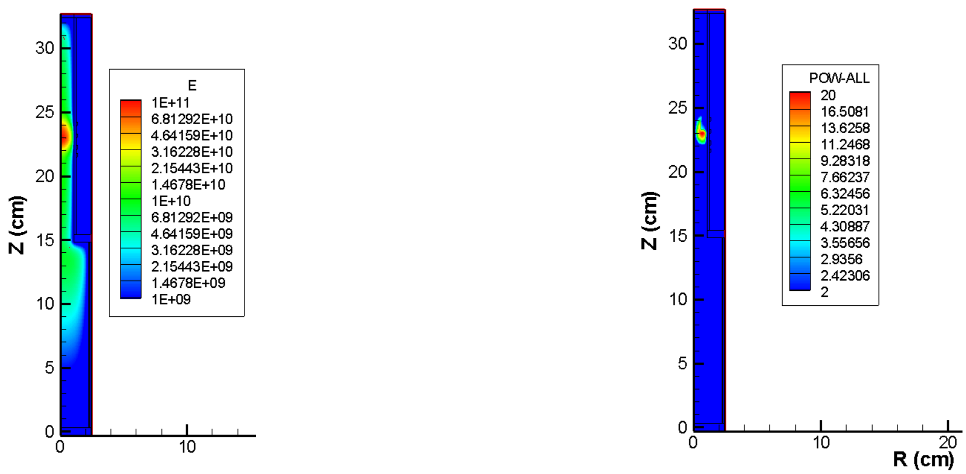


Figure 24: Contours plots depicting a) electron density and b) total power deposited to plasma at 50W

a. b.



As can be seen from Figure 24 above, at 50 W, both the neutral gas (and wall) tempertaure and the electron density are lower than 300 W. However, the electrons penetrate almost the same distance into the tube as with 300 W and thus the substrate is still desirably exposed to the plasma even when there is no flow. Using a similar calculation as Eqn. 1 above, we can determine that the total power transferred to the plasma is around 40 W, given a power deposition of 20 W/cm^3. Since this equation is just an approximation, 40 W is considered close enough to the source power of 50 W.

It can thus be concluded that for a power range between 50 – 300 W, the substrate will lie in the plasma range and thus be exposed to the plasma continually during the reaction process.

It is stressed however that the above modeling procedure was carried out for argon gas since it is the easiest to model. The actual system uses methane and hydrogen for the reactant gases which have an increased number of excited states, ions, electrons and thus reaction mechanisms, increasing the complexity considerably.
Furthermore, the modeling has been carried out for a 4mm diameter inductive coil with 4 windings along the tube. Since it is likely that the coil incorporated in the final design would actually be a commercially available ⅛ inch (6mm) coil, the above results for the temperaure ranges will vary, with an increased probability of the system experiencing higher temperatures than stated.

Modeling analysis was carried out for the same conditions but with different coil diameter of approximately 6mm and 50 W power supply. The plotted results are shown in Appendix I. The average wall temperature at 50 W is estimated at 50°C (323 K), and the total power deposited is higher at 38 W/cm^3. The electron denisty penetrates to the same extent into the chamber as with the 4mm coils. Since modeling data is currently unavailable for 300 W, it is unclear on how much the temperature varies with the 6 mm coils and thus no upper bounds on the temperature have been determined yet.

The total increase in temperature is anticipated to be around 150 – 200 K. As a safety precaution, fans will be positioned along the tube to cool the tube exterior and the components connected to it, in case of a steep rise in temperature.

Vacuum Pump
An important item for the system is the vacuum pump, which creates favorable conditions to generate plasma. The critical customer requirements for the pump are the throughput, at 100 standard cubic centimeters per minute (sccm) and the pressure, at 1 mTorr. Below the formula for throughput is shown, which relates the throughput, pressure and pumping speed.

$$Pumping\ speed = \frac{Throughput}{Pressure} \qquad \text{(Eq. 2)}$$

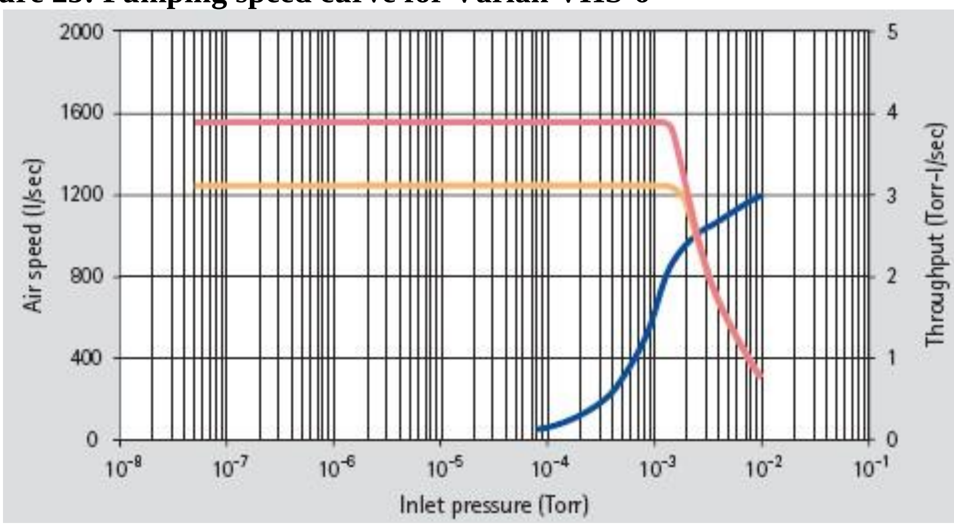
To try to reduce the cost of a pump, a secondary pressure of 5 mTorr was prescribed by the customer. Below in Table 3, for a given throughput and pressure the required pumping speed is shown.

Table 3: Calculation of Pumping Speed with Throughput and Pressure

Throughput (sccm)	Pressure (mTorr)	Pumping speed (L/s)
100	1	1267.817
100	5	253.75

Both of these pumping speeds require diffusion pumps. For a pressure of 1 mtorr the pump that would be recommend would be a Varian VHS-6 and for a pressure of 5 mtorr the pump that would be recommend would be a Varian VHS-4. A pumping speed curve for the VHS-6 is shown below in Figure 25.

Figure 25: Pumping speed curve for Varian VHS-6



For all diffusion pumps, an example of which can be seen above, once the pump gets into its working pressure range, the throughput is constant for pressures lower than the threshold pressure. This can be useful for the system, because if the pressures needed for better plasma generation, or carbon nanotube growth, are lower than 1 mTorr, these types of pumps can expand the experimentation range.

A drawback to these pumps is that the price is higher than mechanical pumps. Another drawback is these pumps need a backing pump to reduce the pressure to help the pump work efficiently. This increases the cost of the pump, since you need to purchase two pumps to in order to achieve optimum conditions.

Dimensions on quartz tube length

Since the placement of the coil was restricted to region near the center or not too close to the two ends of the tube, different tube lengths needed to be modeled to verify their effect on the plasma's ability reach to the substrate. This is because the two ends of the tube consist of O-rings which cannot withstand the high temperatures that they might experience of near the coil. For simplicity, the chamber was modeled as a cylinder with the quartz tube connecting to it on one end. Initially, modeling was carried out with a tube length of 30 cm. However, this proved to be ineffective, with most of the plasma formed being concentrated at the end of the tube farthest from the chamber. Thus the tube was made shorter and modeled at 15 cm. This seemed to provide the appropriate length for the plasma which was now able to penetrate more than half of the inner chamber as show in Fig 23 above. Conversely, the diameter of the tube was provided by our sponsor as 2.5 cm. Since the quartz tube was connected to the chamber by means of an O-ring, we believed that making the tube any shorter would be undesirable, as it would bring the inductive coil closer to the chamber and potentially cause the O-ring experience a temperature higher than its service temperature of 250 – 300°C.

9.6.2 Material Selection

Table 4 below lists the relevant properties for 304L stainless steel and fused quartz as obtained from CES Edupack.

Table 4: Material properties for quartz and stainless steel

Property	**Quartz**	**Stainless Steel**
Max. Service Temperature	1100 – 1400 °C	750 – 925 °C
Transparency	Transparent	Opaque
Chemical Stability	Very good	Very good
Outgassing Rate	7.35×10^{-9} torr-L/(cm^2-s)	5×10^{-8} torr-L/(cm^2-s)
Conventional machining energy	-	10.6 – 11.7 MJ/kg

The material selection for our design was restricted to the use of quartz and stainless steel by our sponsors to minimize the reactivity of the plasma with the inner chamber walls, the tube and the substrate holder. As seen from the table above, quartz and stainless steel make ideal choices for containing the plasma due to their high service temperatures and chemical stability i.e. they are non-reactant with the hydrocarbon source gases (methane and hydrogen) used. In addition, both materials have very low outgassing rates and thus do not add substantially to the plasma composition nor do they contribute additional pressure (outgassing is defined as the quantity of gas formed from a unit area of the material surface per unit time**).**

9.7 Failure and Safety Analysis

A Failure Mode and Effect analysis (FMEA) was carried out to identify the potential failures in our system, the effect of the failures and possible preventive actions for the failures. We used the DesignSafe software to carry out the FMEA analysis and the reports generated are shown in Appendix J. The primary areas of concern arose from the electrical (wiring), environmental (gas evacuation routes), chemical (presence of reactant gases) and mechanical (fatigue) aspects of our design. The table below provides a list and a brief explanation of the failure modes and safety concerns in our design:

Table 5: Failure mode identification and causes

Hazard Category	*Failure type/ Safety Concern*	*Cause of failure*
Mechanical	Failure in the O-ring	The temperature in the quartz tube could exceed the maximum service temperature of the O-ring, leading to failure
Fluid Pressure	Vacuum	Could be a possible failure cause since improper chamber evacuation would negatively affect CNT growth.
Electrical	Live parts	A major safety concern since the inner chamber is equipped with bare wires and a500 V DC bias between the electrodes
	Lack of grounding	A major safety concern since carelessness in grounding the chamber could lead to electrocution
	Arcing	Undesirable, but arcing could occur between the substrate and the electrode due to the 500V DC bias and the presence of plasma, producing very high temperatures
	Improper Wiring	Could lead to an inefficient system, if not harm the user. Improper wring could result from the electrodes not being properly connected to the power supply or from the power supply not being matched to the plasma load resistance.
	Power supply interruption	Not a major safety or failure issue, but a power break out would require the process to start again, thus involving the spending of more time and resources.
	Coil windings around quartz tube	Could pose a risk of electrocution if touched accidentally, or also provide a source of RF interference with other lab equipment. Needs to be properly insulated with coil insulation and a faraday's cage.
Human factors	Deviations from safe work practices	The primary concern in this area included failure to wear gloves while handling the CNTs and perform standard safety procedures while handling electrical circuits.
Fire and explosions	Hot surfaces	A safety concern involving the high temperatures of the tube and the chamber, which might lead to burns or scalds.
	Flammable gas	Hydrogen is one of the reactant gases used in the process and is also highly flammable. Thus, any leaks or the presence of air in the system can pose a potential fire hazard.
Inadequate egress	Evacuation Routes	This was another safety concern, since any loose connection or leaks in the evacuation routes for the reactant gases and the plasma can raise serious environmental concerns.
Temperature	Severe heat	The formation of plasma releases a high amount of energy and thus could lead to the system components failing faster due to fatigue than their normal lifecycle.
Environmental & Biological	Asphyxiants	Overexposure to methane could be hazardous as it acts as an asphyxiant and thus any leaks have to be taken care of as soon as possible.
Chemical	Reactions with chemicals	This could lead to a possible failure mode and a serious safety concern. If the chamber was accidentally opened during its operation, since the process involves continual reaction

		between the reactant gases and the substrate
Chemical gases	Helium	A safety concern, as acute or chronic respiratory conditions may be aggravated from overexposure to this gas. Thus, regular leak checks must be conducted.
	Hydrogen	As explained previously, hydrogen is a flammable gas and poses a fire hazard
	Methane	As explained previously, methane acts as an asphyxiant and is flammable thus posing the risk of a fire or an explosion, in case of leaks.
Biological	Eye contact	A safety concern since in the case of leakage, methane causes eye irritation
	Skin Contact	A safety concern since in the case of leakage, methane causes skin irritation
Radiation	High speed electrons	Breakdown of reactant gases creates ions and electrons and thus cause radiation in the tube. This can cause unwanted interference with other equipment in lab.

As can be seen from the above table, most of the safety concerns arise from leaks in the tubing connections and improper wiring in the electrical connections. These issues could not be addressed directly in our design and thus we recommend double checking all tubing and electrical connections and testing for leaks on a regular basis to prevent most of the above concerns. Another major failure concern arose from the fact that the temperatures reached by the system could not be accurately predicted by the modeling. It is anticipated that the system would reach temperatures higher than those determined from modeling and this is a primary concern for the O-rings connecting the quartz tube to the chamber and gas tubing. To address this issue, a temporary solution of placing muffin fans along the length of the tube and the coil has been decided. This is a relatively easy and inexpensive procedure and highly effective in reducing the system temperature. However, as a long-term consideration, we would recommend the use of a water jacket or a cooling fin that would cool the quartz tube and the coil.

FMEA analysis was performed for all of the above safety concerns, and failure modes and a risk level report was developed, along with possible considerations for reducing the risk level for each (refer to Appendix J).

9.8 Environmental Analysis

Simapro software was used to carry out our analysis for the environmental impact of our design. Figure 26 below shows the plot for the total raw, air, water, waste and soil emissions for 2300g of 304 L stainless steel and 200g of glass (this was used since it was the closest approximation to quartz in the software). As can be seen Stainless steel has the maximum raw emission at approximately 8000g followed by glass at 4000g. Other emissions are negligible when compared to the total raw emission.

Figure 27 indicates the impact of stainless steel and quartz manufacturing to the mineral content, land use, acidification, eco-toxicity, ozone layer, radiation, climate change, resp. inorganics and organics, and finally carcinogens. As can be seen, stainless steel is the main contributor to most of the impacts. However, to reduce this impact on the environment to this extent would require using a different material for our chamber which presents a challenge, since stainless steel is easily available, cheap and highly suited for non-reactive conditions and machinability. It also has the potential to withstand plasma, which makes it in our case, highly desirable.
Finally, Figure 28 and 29 show that manufacturing stainless steel and quartz has a higher impact on natural resources than on human health. This is in accordance with the raw emission figures above for both quartz and stainless steel.

Figure 26: Total emissions for quartz and stainless steel

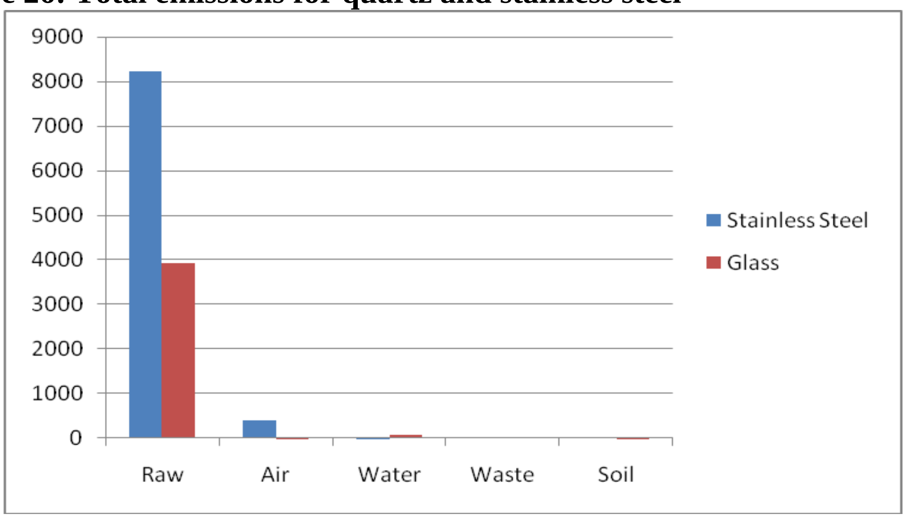


Figure 27: Impact of stainless steel and quartz

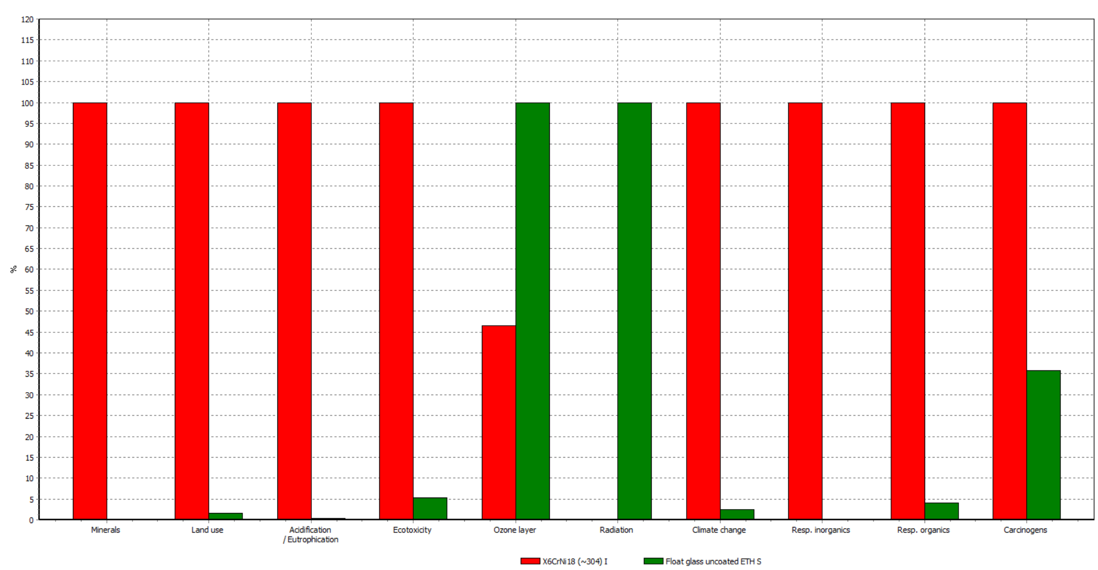


Figure 28: Impact on human health, ecosystem quality and resources

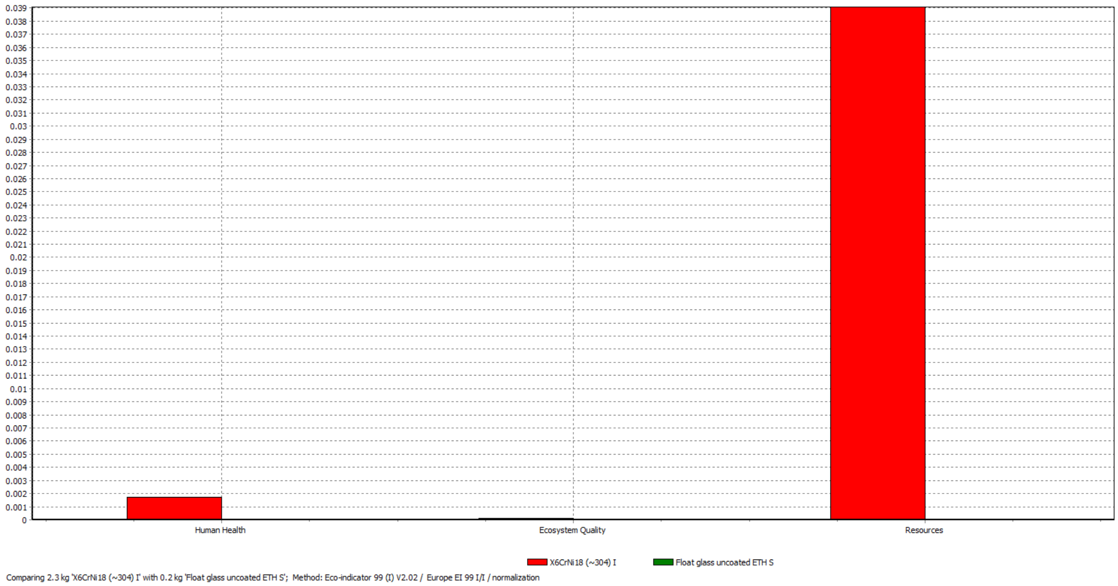
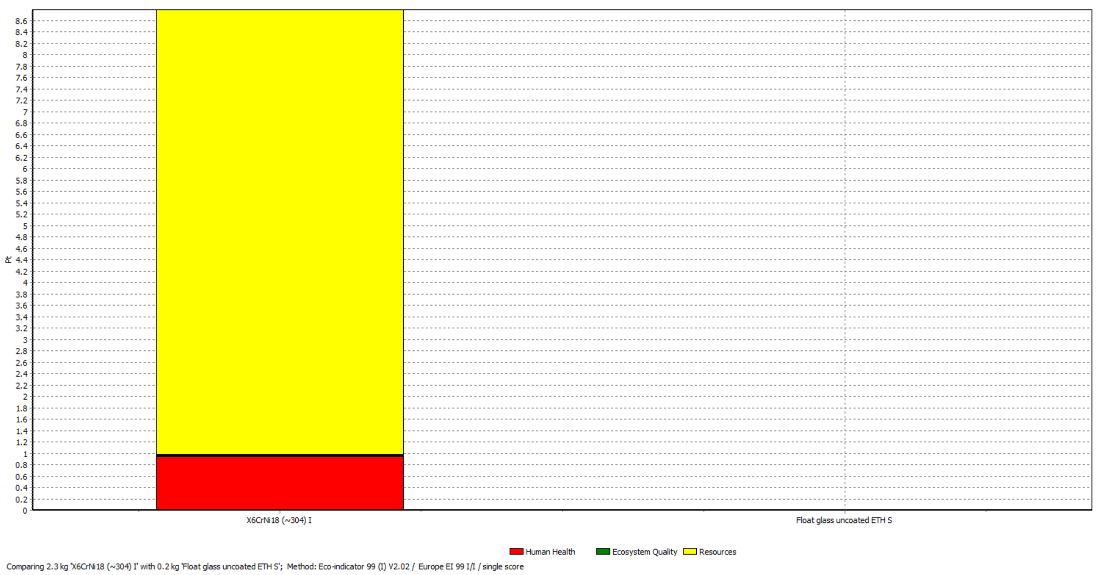


Figure 29: Impact on human health, ecosystem quality and resources



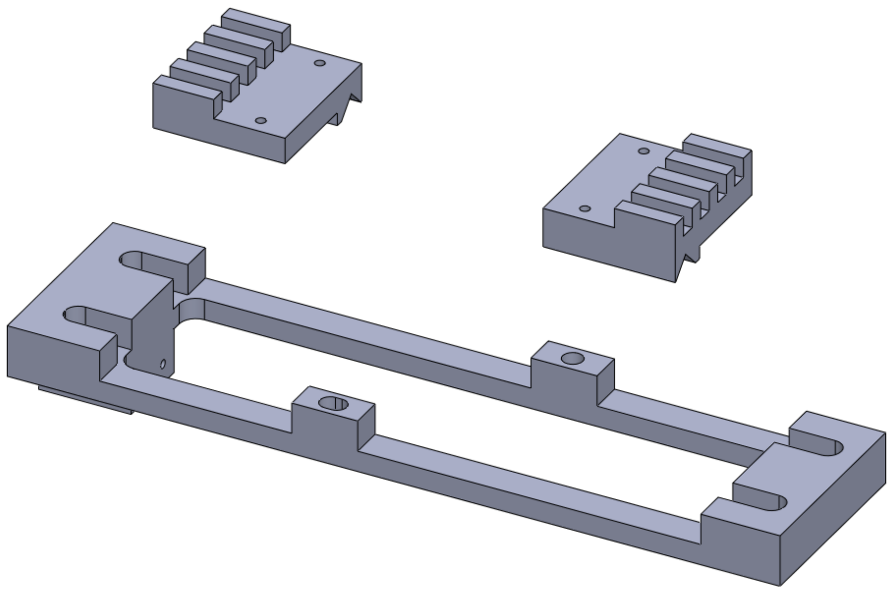
10.0. Manufacturing and Assembly Plan

This section disseminates the steps taken in manufacturing the PECVD system. It explains which components will be professionally machined, machined by the waterjet, and which components are being purchased. It also includes the assembly process used to build the final prototype.

10.1 Outsourced Components

Due to the time constraints as well as the project team's limited machining ability, there are three components that will be professionally machined. These components are the two heat sinks and the system tray, which are shown below in Figure 30. Appendix K shows engineering drawings for outsourced components.

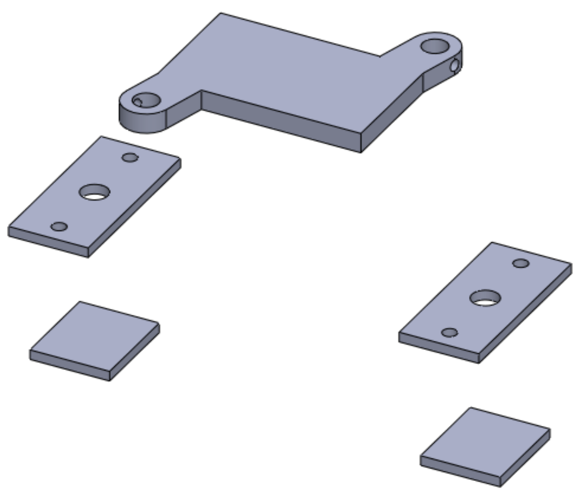
Figure 30: System tray and heat sinks to be professionally machined



10.2 Waterjet machined parts

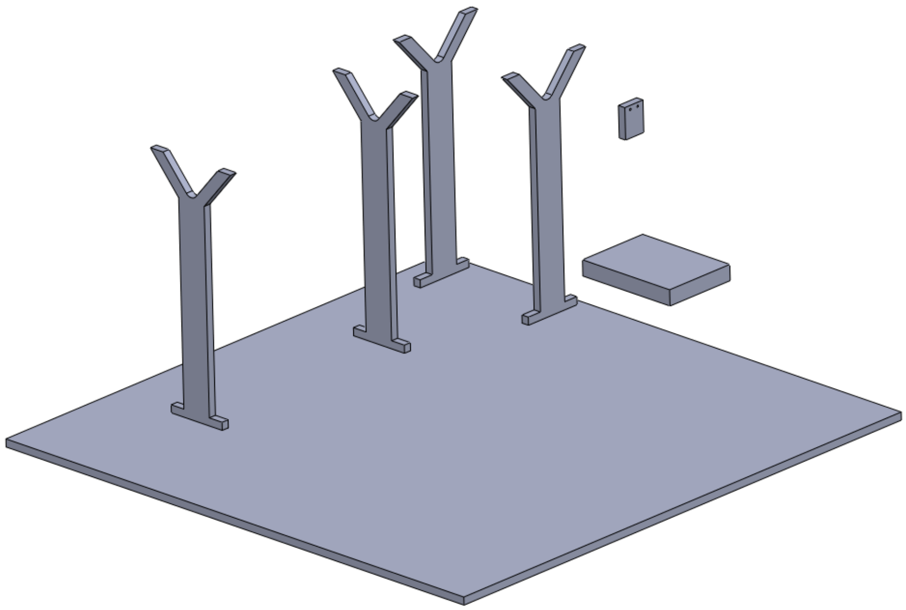
To decrease the machining cost and time, we will use a waterjet machine to make all of the components manufacture in house. As well as minor milling, drilling, and tapping when needed where needed. Below in Figure 31 are the inner chamber components that will be cut using the waterjet.

Figure 31: Inner chamber components to be made with waterjet



The four stands, base plate, guide block plate and internal subassembly connector plate will be made with the waterjet. The stands will have two holes drilled into them to attach them to the base plate. The waterjet will make guide holes in the base plate, which will then be drilled and tapped in order to attach the stands to the base plate. The guide block plate will be cut with the waterjet and the holes will be drilled out and tapped to attach the stand to the guide block. The connector plate will also be cut using the waterjet. Figure 32 below shows the stand components that will be manufactured using the water jet.

Figure 32: Stand components to be cut using the waterjet



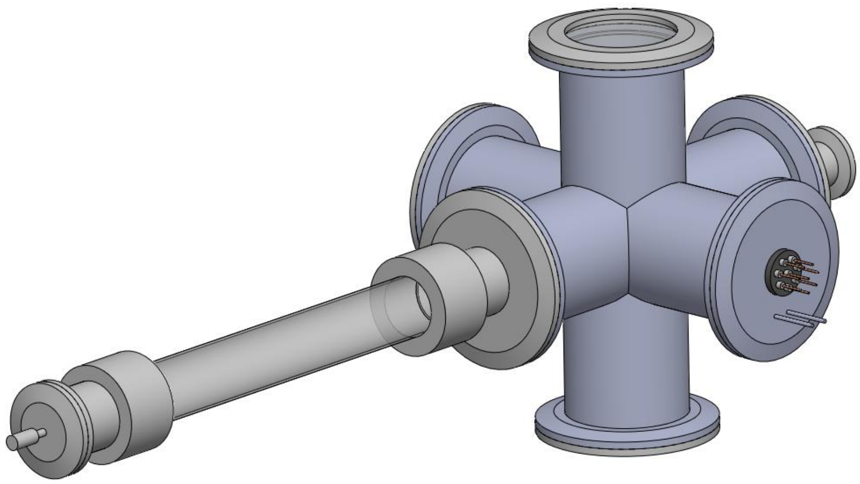
10.3 Purchased Components

Many components in our system are critical for maintaining a vacuum seal are simply too difficult and expensive to have machined. Other components, like a guide block and rails, electronic components, and fasteners make will also be purchased.

10.3.1 Chamber Components

Chamber components are all purchased from Kurt J. Lesker, a vacuum science company specializing in these types of components. All of these components will assemble easily and will fit together properly. Figure 33 below, shows the main chamber components that will be purchased from Kurt J. Lesker.

Figure 33: Chamber components purchased from Kurt J. Lesker



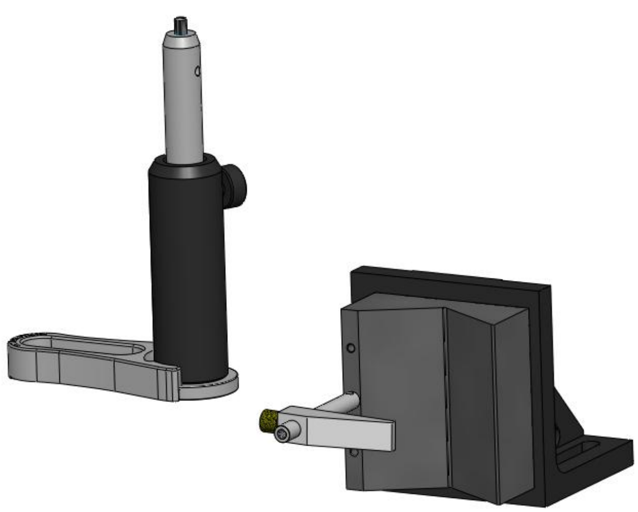
The most difficult manufacturing process for the chamber are two dowels that need to be press-fit through the electric feedthrough flange. This will be a challenging step of the manufacturing process for us and will need to be done very precisely to allow the system tray to attach to the flange, as well as the flange stand to hold up the tray.

Our design calls for four quartz rods and one quartz tube. These components will be purchased from G. Finkenbeiner Inc. and can be seen in Figure 16.

10.3.2 Stand Components

The flange stand is composed of a post holder, post, base adapter and clamping forks which are purchased from Thor Labs. Also purchased from Thor Labs is the V-Mount and right angle plate which are used as to hold the IR sensor. Figure 34 below shows the components purchased from Thor Labs.

Figure 34: Components purchased from Thor Labs



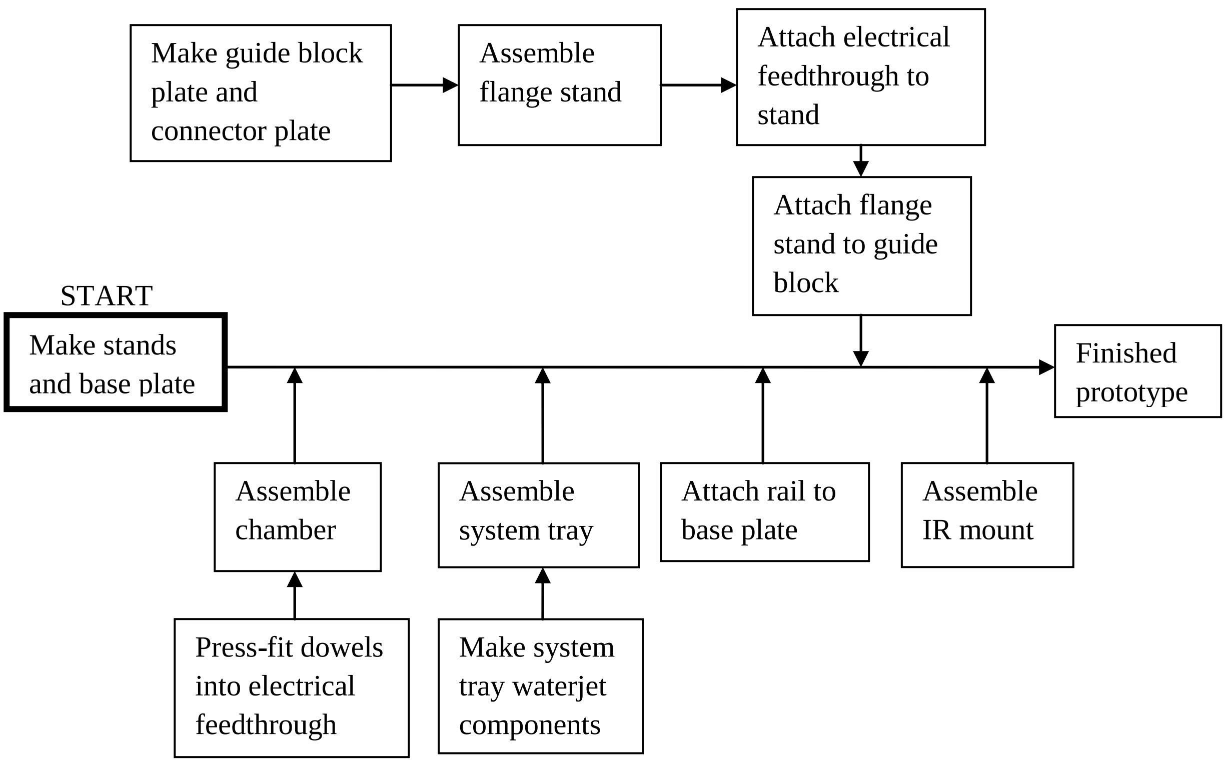
10.3.3 Miscellaneous Components

The IR sensor will be purchased from Exergen. The miscellaneous screws, set screws, dowel pins, and shaft collars will be purchased from McMaster-Carr. Also purchased from McMaster Carr is the guide block and rail. An RF generator and matching network will either be borrowed from the Nanofabrication Lab at the University of Michigan, or will be purchased from PTB sales. A 1/4 inch diameter copper wire will be purchased to make the plasma coil, but a vendor has not been chosen yet.

10.4 Assembly

This section will detail how all of the components are assembled to produce the final prototype. Figure 35 is a flow chart that shows the major steps for assembling the prototype, as well as the general order of completion.

Figure 35: A flow chart illustrating the steps system assembly process



10.4.1 Chamber Assembly

The chamber assembly contains all of the Kurt J. Lesker components as well as the quartz tube. Before the chamber can be fully assembled the dowels must be press-fit into the electrical feedthrough flange. Once this is complete, all of the flanges easily attach to the six-way cross using clamps. The quartz tubing is attached using a quick connect on each end, that are attached using the same type of clamps as the chamber flanges.

10.4.2 System Tray Assembly
The system tray assembly contains the system tray, heatsinks, four quartz rods, the electrode, four plates, a silicon wafer, two silver tipped set screws, and two shaft collars. Two quartz rods lay in the system tray, on top of which the two heatsinks are placed. The silicon wafer is placed between the two heatsinks and the plates are placed on top to secure it. The smaller two quartz rods are placed standing up in the tray where two holes have been made for them. The electrode is slid onto the standing rods and secured with two set screws. The tray is then attached to the press-fit dowel pins with two shaft collars. A picture of the system tray assembly can be seen in Figure 13.

10.4.3 System Tray Stand
The system tray stand contains the rail, guide block, guide block plate, connector plate, stand, and two shaft collars. The electrical feedthrough flange is attached to the stand using the connector plate and secured with two shaft collars. Then the guide block plate is connected to the guide block and the stand, which is attached to the guide block flange. A picture of the system tray stand can be seen in Figure 15.

10.4.4 Miscellaneous Components
The rail and stands will be attached to the base plate by bolts. The IR sensor mount will be attached to a right angle bracket and then bolted to the base plate.

11.0 Usability Analysis

INITIAL SET-UP
- Mount the flange holding the substrate holder to the chamber. Do not place the substrate inside the chamber at this point.
- Mount the quartz tube to the chamber and slide in the inductive coil around the tube. Connect the gas tubing to the inlet of the quartz tube and connect the outlet of the chamber to the pump.
- Slide out the flange containing the substrate holder inside the chamber.
- Connect all the electrical feedthroughs from the flange holder the substrate holder to the electrodes.
- Mount the substrate to the substrate holder and gently slide in the flange into the chamber.
- Complete the electrical connections in the flange by connecting the feedthrough to a DC power supply. Do not turn on the power supply.
- Connect the coil to the matching network, and the matching network to the RF power generator and the automatic matching network controller. Do not turn on the power yet.
- Ensure that all flanges and viewports are in place and that the chamber is completely sealed.

PROCESS SET-UP
- Evacuate the chamber to a low pressure by flowing through the chamber an inert gas such as helium.
- Turn on the gas flow rates to the desired level.
- Turn on the RF generator to the desired power level by setting the forward power option in the generator, and wait till plasma is generated in the quartz tube.
- Ensure that the source impedance (50 Ω) matches the complex load (plasma) impedance by tuning the matching network as necessary, so that maximal power is transferred between the source and load.
- Turn off the generator, the flow rates and the vacuum after a specified amount of time, after which the CNT growth process has reached its final stages or reached a desired level.

- Wait for a few minutes before attempting to dismount the flange and the substrate holder to access the substrate.
- Carefully, disconnect all electrical connections (recommended) and remove the substrate.
- If DC source was to be chosen, then this would have been used instead of the RF generator and an additional step of removing the electrode before accessing the substrate would be encountered.

CLEANING THE CHAMBER AND QUARTZ TUBE

- Ensure that all power supplies, vacuum pumps and gas flow rates are turned off.
- Disconnect the quartz tube from any hosing.
- Remove the coil from the tube.
- Carefully, unmount the quartz tube by removing the flange connecting the tube to the chamber.
- For the chamber, slide off the flange carrying the substrate holder slowly.
- Remove/ disconnect any other flanges or hosing connecting to the chamber, such that the inner chamber can now be accessed and cleaned.

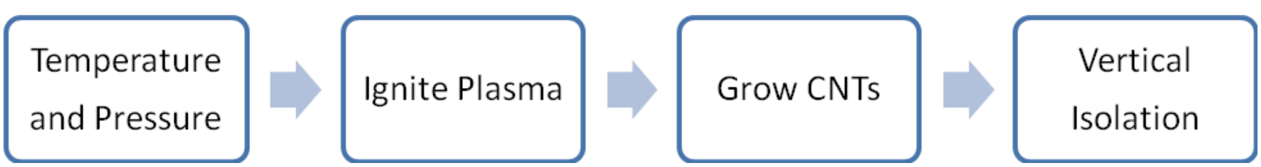
CHANGING ELECTRODES AND THE QUARTZ TUBE HOLDING THE ELECTRODE

- Carefully, slide off the flange carrying the substrate holder.
- Loosen the set-screws holding the electrode on the vertical quartz tubes and slide it off gently.
- Remove the tubes from the substrate holder so that they can be cleaned or replaced as desired.

12.0 Validation Plan

To ensure that the engineering specifications are met, certain experiments must be conducted. The specifications to be tested are listed in Figure 36 below in order of how they will be tested. First, the control of temperature and pressure must be tested to see if the wall temperatures and chamber pressures are too high (leading to failure of some parts). Pressure will be measured using a pressure gauge and the temperature will be measured with a calibrated IR sensor. Analysis of these sensors will tell us if the desired conditions can be met. Second, the creation of plasma must be achieved. This will be proved visually as we will see the plasma ignite through the quartz tube. Next, confirmation that carbon nanotubes are being grown will be achieved by visually seeing black growth on the wafer. Further confirmation of nanotubes growth will be achieved through microscopy using a scanning electron microscope (SEM). Lastly, the SEM will also prove that our system is capable of vertical, isolated nanotubes.

Figure 36: validation plan process



13.0 Risks and Countermeasures

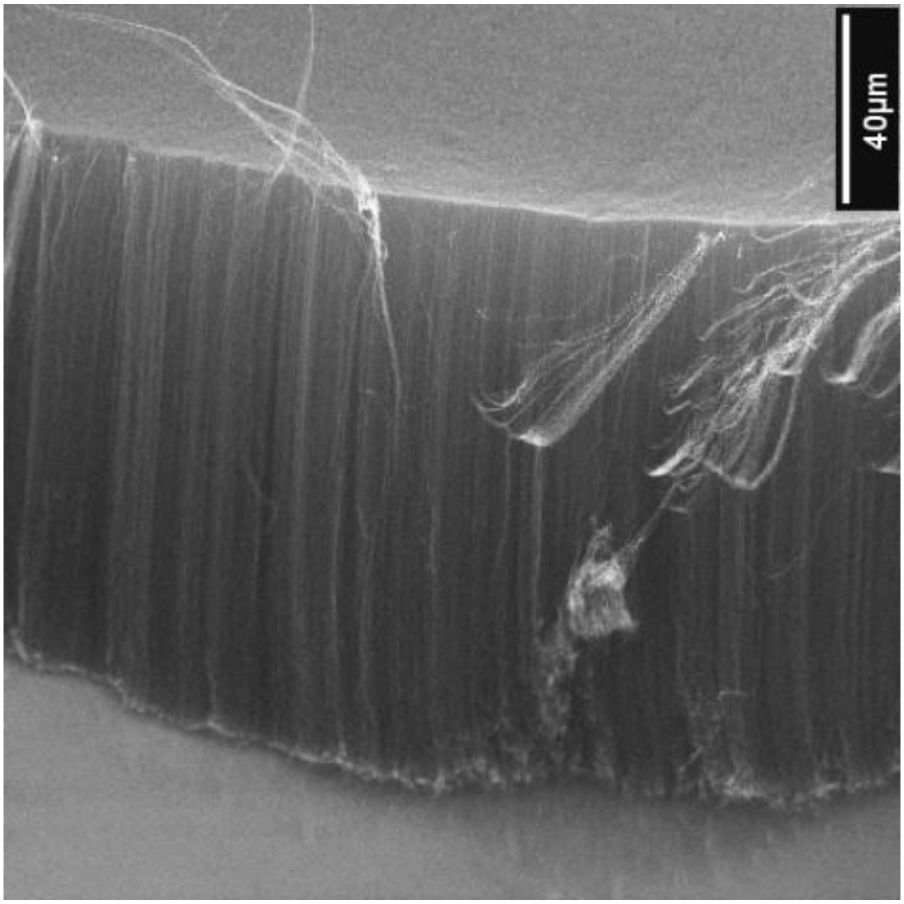
In order to complete the requested work within the three month period allotted a certain level of risk taking was necessary. To assure that none of the risks taken will result in incompletion of the project countermeasures were established. Furthermore, countermeasures were also established to surmount any anticipated potential problems. The identified risks, anticipated problems, and their associated countermeasures are summarized in Table 6 below.

Table 6: Countermeasures to overcome risks and anticipated potential problems

Risk or Anticipated Problem	Countermeasure(s)
Overheating of O-ring used to seal quartz tube	Purchase quartz tube with fixed metal flanges
Using an alternate pump	Rent or borrow a pump capable of the optimum operating conditions

APPENDICES

APPENDIX A: Example of CNT growth



APPENDIX B: Customer Specifications and Engineering Requirements

Customer Requirements and Relative Importance		*Engineering Requirements*	*Target Value*
Capable of vertically aligned CNTs	10	Anode – cathode area(mm)	Minimize
Effective but variable plasma source	9	Power requirement(W)	Minimize
Substrate size	5	Substrate size(mm)	≥ 15 x 15
Adjustable gap between electrodes	7	Adjustable gap size(mm)	10
Precise and non-contact temperature measurement	6	Accuracy of IR temperature Measurement(°C)	± 2
View of substrate	2	Viewing Area(cm)	Maximize
Variable and stable pressure operation	5	Pressure(Torr)	10^{-3}-20
Easy exchange of sample	4	Material compatibility(%)	100
Minimum chamber size	3	Volume of chamber(m^3)	Minimize
Cost	2	Price($)	Minimize
		Temperature Range(°C)	500 - 900
		Flow rate (sccm)	0 – 100

APPENDIX C: Comparison of plasma generation techniques

	Inductively Coupled	DC	Microwave	RF - Triode
Op. Temp	700-900 C	520-750 C	660-1000 C	500-600 C
Op Pressure	1-20 torr	.005 - 4 Torr	10 - 30 Torr	.0075 - .3 Torr
Power	0-200W, 0-300W	50-500W	400-1100 W	50-100W
frequency				13.56 MHz
Plasma gases	hydrogen-metane (80:20) hydrogen-ethylene (80:20)	Ammonia-acetelyne-hydrogen	Methane-hydrogen methane-ammonia ammonia-acetalyne	acetelyne-hydrogen ethalyne-hydrogen
Catalyst		Ti, Ni	Iron Colbalt Nickle	Iron, Ni
gas flow rate	20-100 sccm	80 sccm NH4-30:70 C2H2 sccm	50 - 240 sccm	1 sccm-9 sccm
tube dia	6-30 nm	100nm-microscale	30 nm	10-20nm
tube length			12 - 100 um	2-10 micro m
Substrate		Si	Si, Si/Pt, 25x50 mm Alumina, 2" Si,	2” Si & glass substrate
Growth time			2 - 45 min	2 hr
Min Pressure req.	10^-5 Torr	10^-5 Torr	10^-5 Torr	10^-5 Torr

APPENDIX D: PAIRWISE COMPARISON

Customer Specification	Essential?	PAIRWISE COMPARISONS										TOTAL	WEIGHT
View of substrate	X	0	0	1	1							2	0.2
Substrate size	X	1				1	0	1				3	0.3
Easy exchange of sample	X		1			0			1	1		3	0.3
Minimum chamber size	X			0			1		0		1	2	0.2
Cost	X				0			0		0	0		0
Adjustable gap between electrodes	✓											10	1
Capable of vertically aligned carbon nanotubes	✓											10	1
Effective but variable plasma source	✓											10	1
Precise and non-contact temperature measurement	✓											10	1
Variable and stable pressure operation	✓											10	1

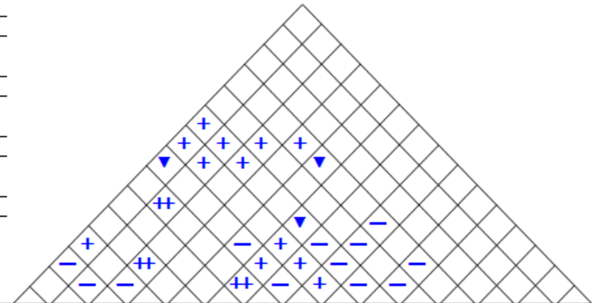
APPENDIX E: QFD

Title: DESIGN OF A PLASMA ENHANCED REACTOR

Author: Janani, Kyler, John, Bryan

Date: 9/17/2008

Notes: Designing a plasma enhanced chemical vapor decomposition reactor for the growth of carbon nanotubes on a silicon substrate



Row #	Max Relationship Value in Row	Relative Weight	Weight / Importance	Demanded Quality (a.k.a. "Customer Requirements" or "Whats") / Quality Characteristics (a.k.a. "Functional Requirements" or "Hows")	1 Pressure	2 Temperature Range	3 Power Requirement	4 Flow Rate	5 Anode/Cathode Area	6 Viewing Area	7 Accuracy of IR Temp Measurement	8 Volume of chamber	9 Material capability	10 Price	11 Adjustable gap size	12 Substrate Size	13	14	15
				Direction of Improvement: Minimize (▼), Maximize (▲), or Target (x)	X	X	▼	X	▼	▲	X	▼	X	▼	X	▲			
1	9	8.2	5.0	Substrate size of 15x15 mm^2					Θ	▲		O		▲		Θ			
2	9	16.4	10.0	View of substrate					▲	Θ		▲	▲	▲	▲	▲			
3	9	9.8	6.0	Precise and non-contact temperature measurement		Θ			▲	▲	Θ			O		▲			
4	9	4.9	3.0	Minimize Chamber size	Θ			▲	Θ	▲		Θ	O	Θ	O	O			
5	9	16.4	10.0	Capable of vertically aligned carbon nanotubes	Θ	Θ	Θ	Θ			▲				Θ				
6	9	3.3	2.0	Low Cost		▲	O			O	O	O	Θ	Θ	▲	▲			
7	9	8.2	5.0	Variable and stable pressure operation	Θ			O		▲		O		O					
8	9	11.5	7.0	Adjustable gap between electrodes								O		O	Θ				
9	3	6.6	4.0	Easy exchange of samples						▲		O		▲	O	▲			
10	9	14.8	9.0	Effective and variable plasma source	Θ	Θ	Θ	Θ	▲					O	Θ				
				Target or Limit Value	10^2 - 20 Torr	500-900 C		0-200 sccm			plus or minus 2 C		100% capable		10 cm	15mm x 15 mm			
				Difficulty (0=Easy to Accomplish, 10=Extremely Difficult)	9	6	5	1	2	6	8	5	2	5	8	6			
				Max Relationship Value in Column	9	9	9	9	9	9	9	9	9	9	9	9			
				Weight / Importance	398.4	372.1	290.2	309.8	159.0	195.1	114.8	173.8	60.7	237.7	437.7	124.6			
				Relative Weight	13.9	12.9	10.1	10.8	5.5	6.8	4.0	6.0	2.1	8.3	15.2	4.3			

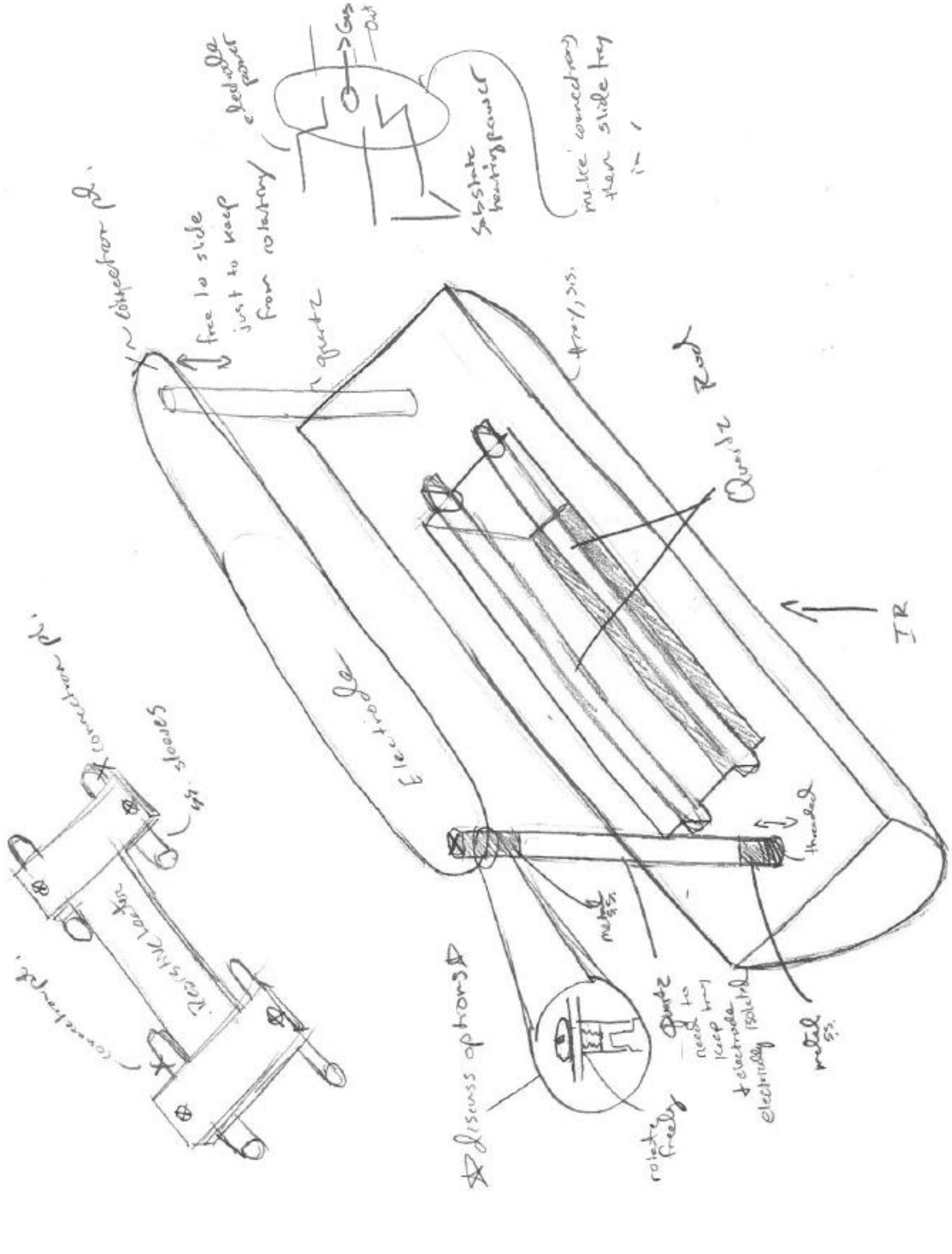
Competitive Analysis (0=Worst, 5=Best)

RF Plasma Mode	DC Plasma	Inductively Coupled Plasma	Microwave Plasma

0 1 2 3 4 5

Legend		
Θ	Strong Relationship	9
O	Moderate Relationship	3
▲	Weak Relationship	1
++	Strong Positive Correlation	
+	Positive Correlation	
—	Negative Correlation	
▼	Strong Negative Correlation	
▼	Objective Is To Minimize	
▲	Objective Is To Maximize	
X	Objective Is To Hit Target	

APPENDIX F: Submodule concept sketch



APPENDIX G: BOM

Item #	Vendor	Part#	Item name	Function or module	Leadtime	Qty	Unit (each, box, etc.)	Unit price	TOTAL
1	Lesker	QF50-200-6X	6-way cross	Chamber	In Stock	1	1	306	306
2	Lesker	QF50-200-VP	viewport	Chamber viewports	In Stock	3	1	106.25	318.75
3	Lesker	QF50XVC100	KF50 quick connect	Chamber side tube connection	In Stock	1	1	78.2	78.2
4	Lesker	QF25XVC100	KF25 quick connect	Inlet side tube connection	In Stock	1	1	68	68
5	Lesker	QF25X4SWG	KF25 swagelok adapter	Inlet swagelok adapter	In Stock	1	1	51	51
6	Lesker	QF50-200-CHP	KF50 lever clamp	flange sealing	In Stock	6	1	17	102
7	Lesker	QF25-100-CHP	KF25 lever clamp	attach inlet to quartz tube	In Stock	1	1	14.31	14.31
8	Lesker	QF50-200-SRV	KF50 center ring	seal between flanges	In Stock	6	1	11.9	71.4
9	Lesker	QF25-100-SRV	KF25 center ring	seal between flanges	In Stock	1	1	6.8	6.8
10	Lesker	EFT0083038C	weldable flange and feedthrough	electric feedthrough	1 week	1	1	188.06	188.06
11	Lesker	QF50XQF16	Nipple reducing QF50 to QF16	Attach outlet hose to chamber	In Stock	1	1	42.5	42.5
12	McMaster-Carr	98381A428	steel pin	Press-fit pin, attach boat to flange	In stock	2	1	3.4	6.8
13	McMaster-Carr	6462K71	shaft collar	Hold pins onto boat and block	In Stock	4	1	2.46	9.84
14	McMaster-Carr	99934A140	soft tip set screw	Hold up electrode on rods	In Stock	2	5	5	10
15	McMaster-Carr	91830A401	thumb screw	hold heating wafer to heat sinks	In Stock	2	1	4.38	8.76

16	McMaster-Carr	3249K3	Lockable Plain Bearing Guide Block	Moving boat into/out of chamber	In Stock	1	1	71.84	71.84
17	McMaster-Carr	9867K13	Guide Rails	Moving boat into/out of chamber	In Stock	1	500 mm	65	65
18	Thor Labs	VC3	Fixed V-Clamp	IR Sensor Holder	In Stock	1	1	42.3	42.3
19	Thor Labs	PH3-ST	3 in Post Holder	Height Adjustment	In Stock	1	1	8.27	8.27
20	Thor Labs	TR3	OD1/2" x 3" post	Height Adjustment	In Stock	1	1	5.42	5.42
21	Thor Labs	BE1	Pedestal Base Adapter - Imperial	Stand	In Stock	1	1	9.1	9.1
22	Thor Labs	CF125	Small Clamping Fork	Stand	In Stock	1	1	8.3	8.3
23	Exergen	IRt/c.4ALF LoE	IR Sensor	Temp. Sensor	In Stock	1	1	735	735
24	ENI	ENI ACG-5 ENI 5D	RF generator + matching network		In Stock	1	1	5000	
25	Varian	VHS-6	Vacuum Pump		In Stock	1	1	7000	

APPENDIX H: Contour plots for the temperature of Argon in its neutral and excited states at 50 W and 300 W

Fig. 31: Contour plots depicting temperatures of a) Neutral Gas (T-AR), b) Excited state of neutral gas Ar* (T-Ar*), and c) Excited state of neutral gas Ar (T-Ar**) at 50 W**

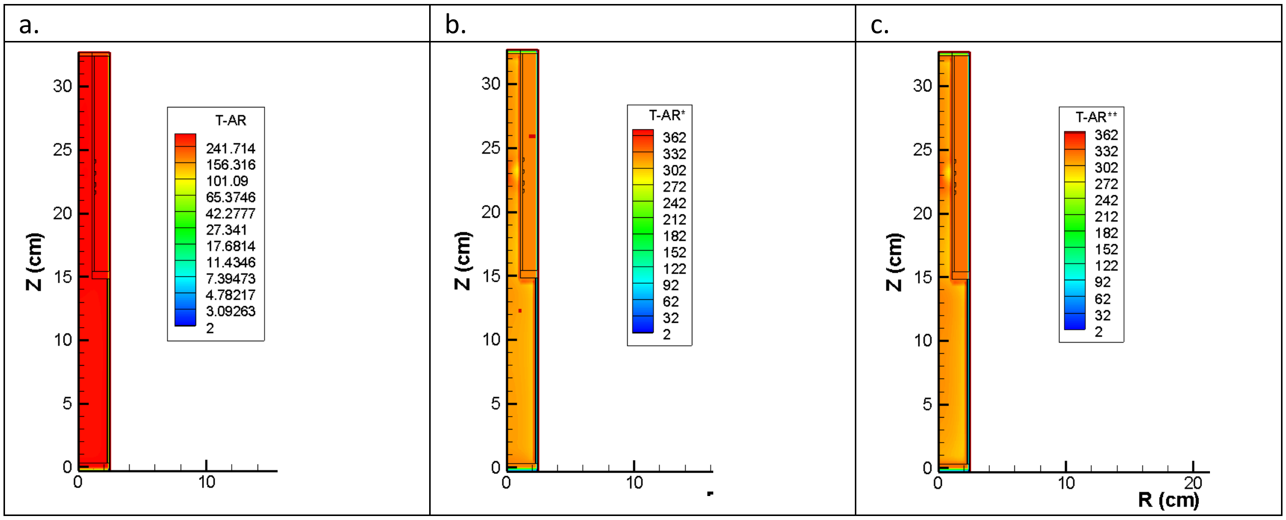
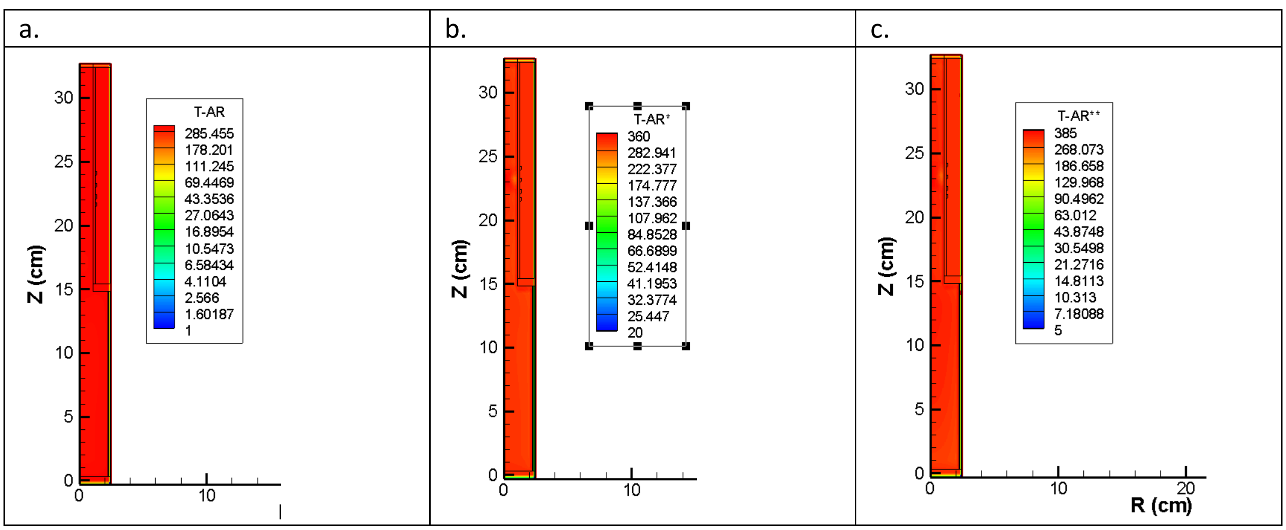


Fig. 32: Contour plots depicting temperatures of a) Neutral Gas (T-AR), b) Excited state of neutral gas Ar* (T-Ar*), and c) Excited state of neutral gas Ar (T-Ar**) at 300 W**



APPENDIX I: Contour plots for the temperature, electron density and total power deposited to the plasma at 50 W, with 6mm coils

Fig. 33: Contour plots depicting temperatures of a) Neutral Gas (T-AR), b) Excited state of neutral gas Ar* (T-Ar*), and c) Excited state of neutral gas Ar (T-Ar**) at 50 W**

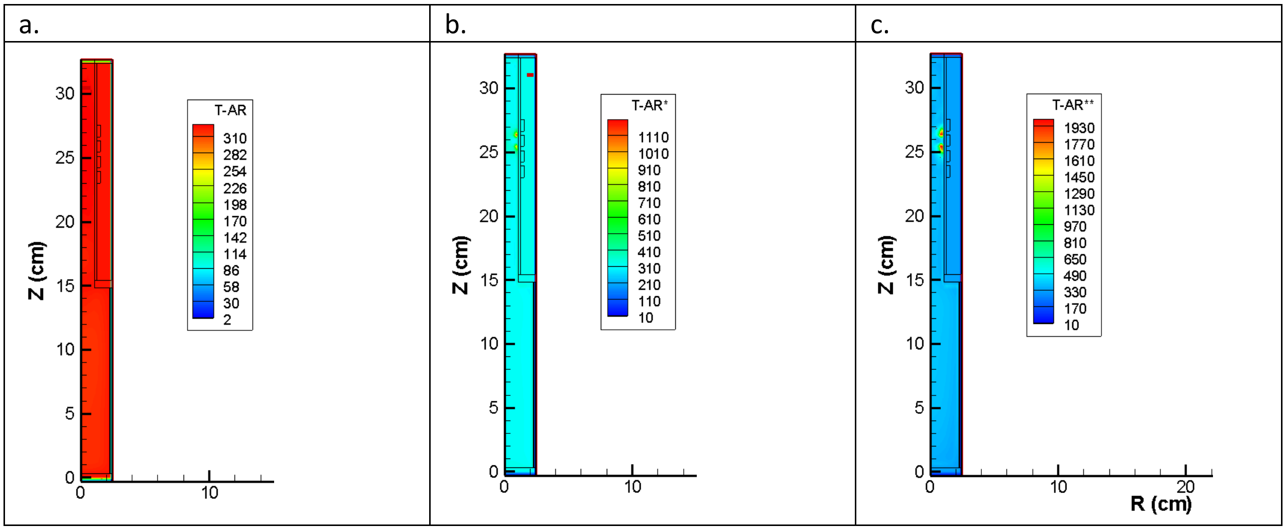
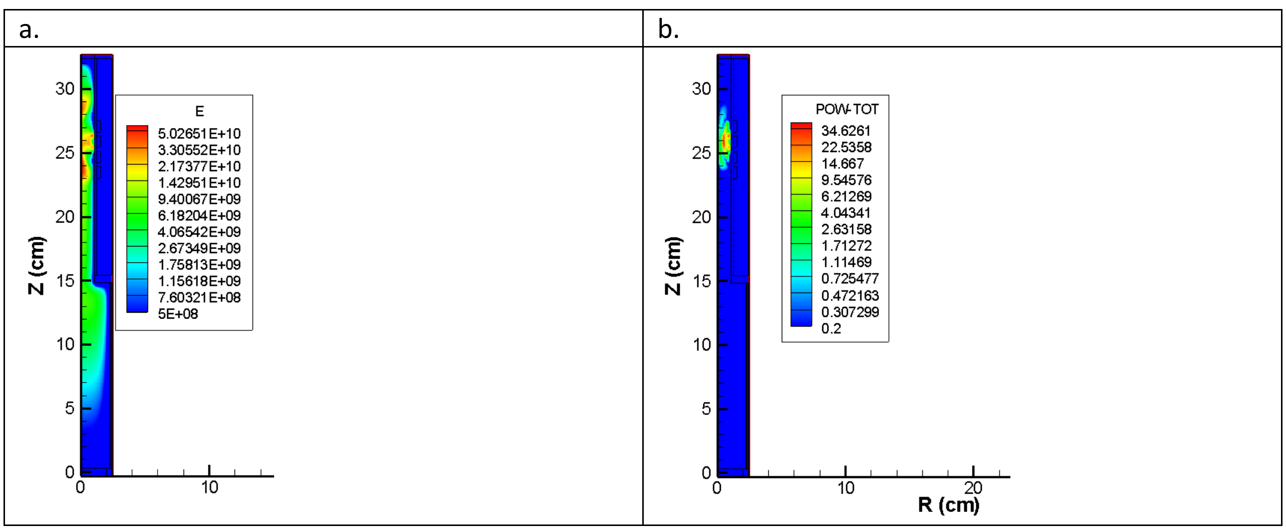


Fig. 34: Contour plots depicting temperatures of a) electron density (E), and b) total power deposited to load at 50 W



APPENDIX J: FMEA Analysis

Risk Level Report

Application:	PECVD system	Analyst Name(s):	Janani Viswanathan, Bryan yamasaki, Kyler Nicholson, John taphouse
Description:	ME 450 plasma Enhanced Chemical Vapor Deposition System for growing carbon nanotubes	Company:	University of Michigan
Product Identifier:		Facility Location:	Mechanosynthesis Lab
Assessment Type:	Detailed		
Limits:			
Sources:			

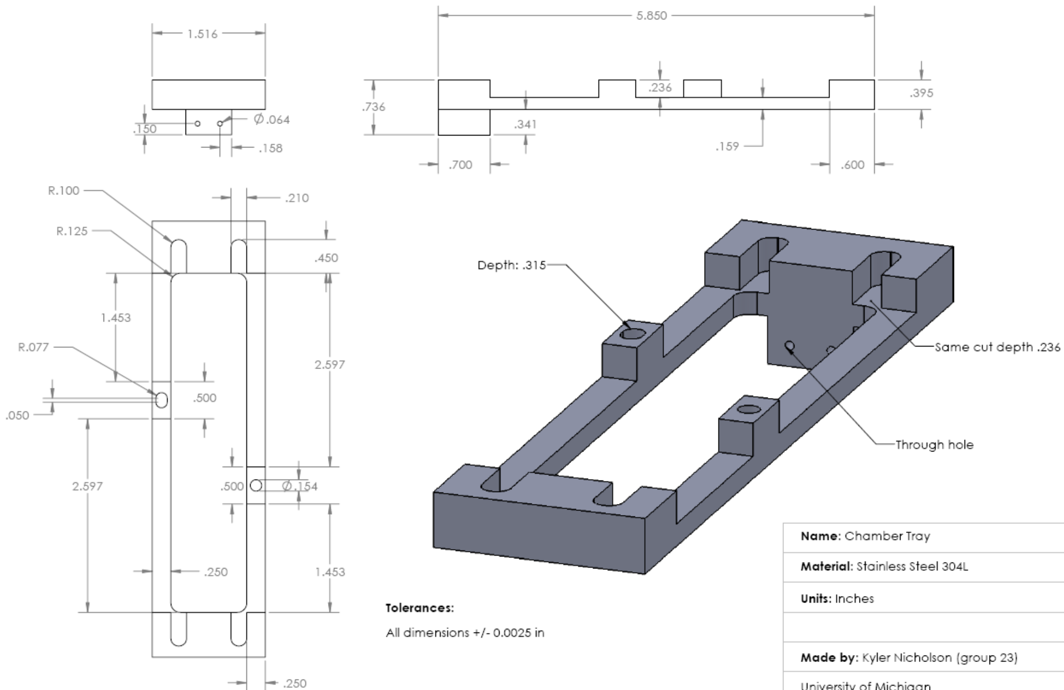
Guide sentence: When doing [task], the [user] could be injured by the [hazard] due to the [failure mode].

Final Assessment Severity Exposure Probability	Risk Level	User / Task	Hazard / Failure Mode	Risk Reduction Methods / /Comments	Initial Assessment Severity Exposure Probability	Risk Level	Status / Responsible / /Reference
Minimal Occasional Unlikely	Low	All Users All Tasks	mechanical : fatigue O-ring - Tempertaure can exceed O-ring specification	Replace O-rings	Serious Occasional Possible	High	
Minimal None Negligible	Low	All Users All Tasks	electrical / electronic : energized equipment / live parts 300W Power supply, bare wires inside chamber with Dc current, 500V Dc bias inside chamber	Make sure all power supplies are turned off before handling	Catastrophic Remote Possible	High	
Minimal None Negligible	Low	All Users All Tasks	electrical / electronic : lack of grounding (earthing or neutral) Chamber might not be grounded properly	Check if all connections are done properly	Catastrophic Remote Possible	High	
		All Users All Tasks	electrical / electronic : shorts / arcing / sparking Between the electrodes and the substrate in the presence of plasma		Slight Occasional Probable	High	
Slight None Unlikely	Low	All Users All Tasks	electrical / electronic : improper wiring Electrodes not connected properly to power supply, power supply not matched to load	Check if all connections made properly	Catastrophic Remote Possible	High	
Minimal None Negligible	Low	All Users All Tasks	electrical / electronic : power supply interruption Power break-out	Turn off power supply	Slight None Possible	Low	

Final Assessment Severity Exposure Probability	Risk Level	User / Task	Hazard / Failure Mode	Risk Reduction Methods / /Comments	Initial Assessment Severity Exposure Probability	Risk Level	Status / Responsible / /Reference
		All Users All Tasks	electrical / electronic : electromagnetic susceptibility				
Minimal Remote Unlikely	Low	All Users All Tasks	ergonomics / human factors : deviations from safe work practices No gloves while handling Carbon nanotubes	Need to wear gloves to handle carbon nanotubes	Slight Remote Unlikely	Low	
Slight None Unlikely	Low	All Users All Tasks	fire and explosions : hot surfaces Inner chamber wall temperature at a high temperature durng process	Allow for a few minutes for chamber to cool down before opening it up.	Minimal None Unlikely	Low	
Minimal None Negligible	Low	All Users All Tasks	fire and explosions : flammable gas presence of hydrogen, methane	Check tubings for leakages and all hose connections	Catastrophic None Unlikely	Moderate	
Minimal None Negligible	Low	All Users All Tasks	fire and explosions : inadequate egress / evacuation routes If connection to evacuation route is loose/ leakage, can cause environmental concerns	Check outlet connections and tubing	Catastrophic Remote Possible	High	
Minimal Remote Unlikely	Low	All Users All Tasks	heat / temperature : severe heat plasma formation	Do not touch chamber during process	Serious Frequent Possible	High	
Minimal None Negligible	Low	All Users All Tasks	environmental / industrial hygiene : asphyxiants methane in case of leaking is an asphyxiant	Check tubings for leakages and all hose connections	Slight Remote Negligible	Low	
Minimal None Negligible	Low	All Users All Tasks	environmental / industrial hygiene : effluent / effluent handling helium, hydrogen, methane flowing through chamber and evacuated	Check tubings for leakages and all hose connections	Minimal Remote Possible	Low	
Minimal None Negligible	Low	All Users All Tasks	chemical : reaction to / with chemicals hydrogen and methane reacting	Do not open chamber during process	Minimal None Negligible	Low	

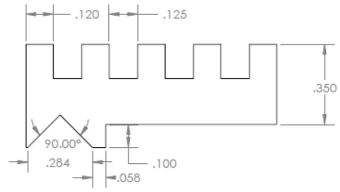
Final Assessment Severity Exposure Probability	Risk Level	User / Task	Hazard / Failure Mode	Risk Reduction Methods / /Comments	Initial Assessment Severity Exposure Probability	Risk Level	Status / Responsible / /Reference
Minimal None Negligible	Low	All Users All Tasks	chemicals and gases : helium Acute or chronic respiratory conditions may be aggravated by overexposure to this gas	Check tubings for leakages and all hose connections, do not open chamber during process	Serious None Negligible	Low	
Minimal None Negligible	Low	All Users All Tasks	chemicals and gases : hydrogen flammable	Check tubings for leakages and all hose connections	Serious Remote Unlikely	Moderate	
Minimal None Negligible	Low	All Users All Tasks	chemicals and gases : methane flammable, At high concentration methane acts as an asphyxiant, fire, explosion	Check tubings for leakages and all hose connections	Serious Remote Unlikely	Moderate	
Minimal None Negligible	Low	All Users All Tasks	biological / health : asphyxiant presence of methane	Check for leakages	Serious Remote Unlikely	Moderate	
Minimal None Negligible	Low	All Users All Tasks	biological / health : eye contact in case of leakage, methane causes eye irritation	Check for leakages, if not wear safety glasses	Serious Remote Unlikely	Moderate	
Minimal None Negligible	Low	All Users All Tasks	biological / health : skin contact in case of leakage, methane causes skin irritation	Check for leakages	Serious Remote Unlikely	Moderate	
Slight None Possible	Low	All Users All Tasks	fluid / pressure : vacuum Improper chamber evacuation might cause system not to work	Make sure not to touch the inner chamber walls or wear gloves, all connections are properly connected/ sealed	Minimal Frequent Probable	High	
Minimal None Negligible	Low	All Users All Tasks	fluid / pressure : fluid leakage / ejection in tubing connections	Check tubings for leakages and all hose connections	Serious Remote Unlikely	Moderate	
Minimal None Negligible	Low	All Users All Tasks	radiation : high-speed electrons breakdown of reactant gases to ions and electrons	Do not open chamber during process	Serious Remote Unlikely	Moderate	

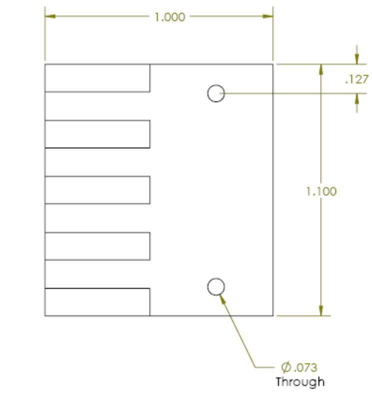
APPENDIX K: Engineering drawings: system tray and heat sinks

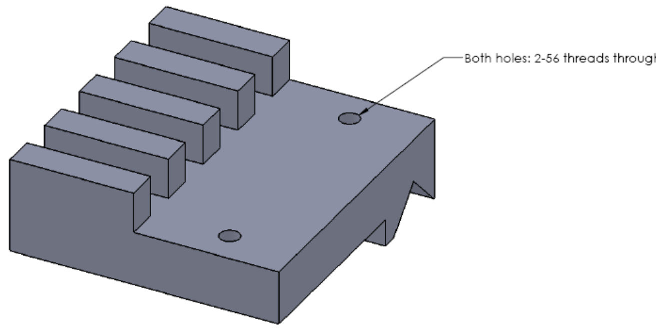


Tolerances:
All dimensions +/- 0.0025 in

Name: Chamber Tray
Material: Stainless Steel 304L
Units: Inches
Made by: Kyler Nicholson (group 23)
University of Michigan
Date: 11/09/2008







Tolerances:

All dimensions +/- 0.0025 in
Angle +/- 5 degrees

Name: Heat Sink
Material: Stainless Steel 304L
Units: Inches
Made by: Kyler Nicholson (group 23)
University of Michigan
Date: 11/09/2008

REFERENCES

[1] Li, J., Ye Q., Cassel A., Ng, H., Stevens, R., Han, J., and Meyyappan, M., 2003, “Bottom Up Approach for Carbon Nanotube Interconnects,” Applied Physics Letters, 85(15), pp. 2491-2493

[2] Delzeit, L., McAninch, I., Cruden, B., Hash, D., Chen, B., Han, J., and Meyyappan, M., 2002, “Growth of Multiwall Carbon Nanotubes in an Inductively Coupled Plasma Reactor,” Journal of Applied Physics, 91(9), pp. 6027-6033

[3] Delzeit, L., Nguyen, C., Stevens, R., Han, J., and Meyyappan, M, 2002, “Growth of Carbon Nanotubes by Thermal and Plasma Chemical Vapor Deposition Process and Applications in Microscopy,” Institute of Physics Publishing: Nanotechnology, 13, pp. 280-284

[4] Mathews, K., Cruden, B. Chen, B., Meyyappan, M., and Delzeit, L., 2002, “Plasma-Enhanced Chemical Vapor Deposition of Multiwalled Carbon Nanofibers,” Journal of Nanoscience and Nanotechnology, 2(5), pp. 475-480

[5] Cui, H., Zhou, O., and Stoner, B., “Deposition of Aligned Bamboo-Like Carbon Nanotubes Via Microwave Plasma Enhanced Chemical Vapor Deposition,” Journal of Applied Physics, 88(10), pp. 6072-6074

[6] Tsia, C., Chen, C., and Wu, L., “Bias Effect on the Growth of Carbon Nanotips Using Microwave Plasma Chemical Vapor Deposition,” Applied Physics Letters, 81(4), pp. 721-723

[7] Qin, L., Zhou, D., Krauss, A., and Gruen, D., 1998, “Growing Carbon Nanotubes Using Microwave Plasma-Enhanced Chemical Vapor Deposition,” Applied Physics Letters, 72(26), pp. 3437-3439

[8] Bower, C., Zhu, W., Jin, S., and Zhou, O., 2000, “Plasma Induced Alignment of Carbon Nanotubes,” Applied Physics Letters, 77(6), pp. 830-832

[9] Bower, C., Zhou, O., Zhu, W., Werder, J., and Jin, S., 2002, “Nucleation and Growth of Carbon Nanotubes by Microwave Plasma Chemical Vapor Deposition,” Applied Physics Letters, 77(17), pp. 2767-2769

[10] Tsia, S., Chao, C., Lee, C., and Shih, H., 1999, “Biased-Enhanced Nucleation and Growth of the Aligned Carbon Nanotubes with Open Ends with Microwave Plasma Synthesis,” Applied Physics Letters, 74(23), pp. 3462-3464

[11] Choi, Y., Shin, Y., Lim, S., Bae, D., Lee, Y., and Lee, B., 2000, “Effect of Surface Morphology of Ni Thin Film on the Growth of Aligned Carbon Nanotubes by Microwave Plasma-Enhanced Chemical Vapor Deposition,” Journal of Applied Physics, 88(8), pp. 4898-4903

[12] Hopwood, J., 1992, “Review of Inductively Coupled Plasmas for Plasma Processing,” Plasma Sources Sci. Technol., 1, pp. 109-116

[13] Conrads, H., Schmidt, M., 2000, “Plasma Generation and Plasma Sources,” Plasma Sources Sci. Technol, 9, pp. 441-454

[14] Hyun, D., Shin, D., Lee, W., and Kim, S., 2008, "Hierarchically Organized Carbon Nanotube Arrays from Self-Assembled Block Copolymer Nanotemplates," Advanced Materials, 20, pp. 2480-2485

[15] Qi, H., Teo, K., Lau, K., Boyce, M., Milne, W., Robertson, J., and Gleason, K., 2003, "Determination of Mechanical Properties of Carbon Nanotubes and Vertically Aligned Carbon Nanotube Forests using Nanoindentation," Journal of the Mechanics and Physics of Solids, 51, pp. 2213-2237

[16] Semet, V., Binh, V., Guillot, D., Teo, K, Chhowalla, M., Amaratunga, G., Milne, W., Legagneax, P., and Pribat, D., 2005, "Reversible Electromechanical Characteristics of Individual Multiwall Carbon Nanotubes," Applied Physics Letters, 87(22)

[17] Li, Y., Mann, D., Rolandi, M., Kim, W., Ural, A., Hung, S., Javey, A., Cao, J., Wang, D., Yenilmez, E., Wang, Q., Gibbons, J., Nishi, Y., and Dai, H., 2004, "Preferential Growth of Semiconducting Single-Walled Carbon Nanotubes by a Plasma Enhanced CVD Method," Nano Letters, 4(2), pp. 317-321

[18] Bell, M., Lacerda, R., Teo, K., and Milne, W., 2006, "Characterisation of the Growth Mechanism During PECVD of Multiwalled Carbon Nanotubes," Topics Appl. Phys., 100, pp. 77-93

[19] Merkulov, V., Guillorn, M., Lowndes, D., and Simpson, M., 2001, "Shaping Carbon Nanostructures by Controlling the Synthesis Process," Applied Physics Letters, 79(8), pp. 1178-1180

[20] Guillorn, M., Simpson, M., Bordonaro, G., Merkulov, V., Baylor, L., and Lowndes, D., 2001, "Fabrication of Gated Cathode Structures Using an *In Situ* Grown Vertically Aligned Carbon Nanofiber as a Field Emission Element," Vac. Sci. Technol., 19(2), pp. 573-578

[21] Guillorn, M., Melechko, A., Merkulov, V., Ellis, E., Simpson, M., Lowndes, D., Baylor, L., and Bordonaro, G., 2001, "Microfabricated Field Emission Devices using Carbon Nanofibers as Cathode Elements," Vac. Sci. Technol., 19(6), pp. 2598-2601

[22] Mayyappan, M., Delzeit, L., Cassel, A., and Hash, D., 2003, "Carbon Nanotube Growth by PECVD: A Review," Institute of Physics Publishing, 12, pp. 205-216

[23] Shiratori, Y., Hiraoka, H., and Yamamoto, M., 2004, "Vertically Aligned Carbon Nanotubes Produced by Radio-Frequency Plasma-Enhanced Chemical Vapor Deposition at Low Temperature and their Growth Mechanism," Materials Chemistry and Physics, 87, pp. 31-38

[24] Show, Y., and Fukuzumi, N., 2007, "Selective Growth of CNT by Using Triode Type Radio Frequency Plasma Chemical Vapor Deposition Method," Diamond and Releted Materals, 16, pp. 1106-1109

[25] Show, Y., Yabe, Y., Izumi, T., and Yamauchi, H., 2005, "Development of Triode Type RF Plasma Enhance CVD Equipment for Low Temperature Growth of Carbon Nanotubes," Diamond and Related Materials, 14, pp. 1848-1851

[26] Yabe, Y., Ohtake, Y., Ishitobi, T., Show, Y., Izumi, T., and Yamauchi, T., 2004, "Synthesis of Well-Aligned Carbon Nanotubes by Radio Frequency Plasma Enhanced CVD Method," Diamond and Related Materials, 13, pp. 1292-1295

[27] Ford, N., 2007, “Plasma Enhanced Growth of Carbon Nanotubes,” Ph.D. thesis, Oregon State University

[28] The United States Patent and Trademark Office website, www.uspto.gov

[29] Dr. John Foster, Director of the Plasma Science and Technology Lab, University of Michigan

[30] Dr. Mark Kushner, Director of the Michigan Institute for Plasma Science and Engineering, University of Michigan

[31] CES EduPak, Granta Design Limited 2008

[32] Michigan Tech, Department of Mechanical Engineering website, www.me.mtu.edu

[33] Varian Inc., Varian website, www.varianinc.com

[34] Kurt J. Lesker VacuCAD Service, www.lesker.com

[35] Thor Labs Support Documentation, www.thorlabs.com

[36] Simapro LCA software, Product Ecology Consultants

[37] Designsafe, Design Safety Engineering, Inc. 2008