# Temperature Programmed Desorption Secondary Ion Mass Spectrometer (TPD-SIMS)

## Final Report

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**2017-2018**

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# DISCLAIMER

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# 1 BACKGROUND

## 1.1 Introduction

The goal of this project is to modify and enhance a Time of Flight Secondary Ion Mass Spectrometer (TOF-SIMS). A TOF-SIMS encompasses an ion source that generates ions, each comprised of two or more atoms, a collection cone which collects the particles and transports them to the flight chamber, and a mass spectrometer that measures the particles and provides the data [1]. This device is used by scientists and researchers to analyze the amount of secondary ions removed from the surface of a sample [2]. As well as understanding problems of surface reaction chemistry through the direct measurement of rate coefficients [3]. One method of accomplishing these goals is called Temperature Programed Desorption (TPD). This technique requires the surface temperature of the sample to be increased to incredibly high temperatures [4]. Within the TOF-SIMS, an ion beam is shot at the surface of the material. When the two collide, secondary ions are released into the vacuum chamber. These secondary ions bounce into the flight chamber where the mass spectrometer is located. The mass spectrometer then collects the secondary ions using different, electric potentials to attract them, and then analyzes the ions to determine different properties and characteristics of the material. Utilization of TPD and SIMS will allow the team to take the measurements needed to fully analyze a sample of second.

The project's objectives are; to keep the chamber at a vacuum state of about 10-7 torr while the system is running, make a sample holder with thermal and electrical insulation from the sample, and to heat the sample to 1450°C at a constant rate of 1°C /s while the sample is under the vacuum. As well as, find a way to measure the temperature from 20°C to 1450°C, and calibrate the electron gun and the sample so the desorbed ions go into the mass spectrometer. Other project objectives are to shield the turbo pump from secondary ions, measure the desorption data with a time resolution of 2 µs. Lastly the mass spectrometer should measure up to 50 mass per charge (m/z).

Dr. Michael Lee is the team's advisor and Sandia National Laboratories is the team's clients and sponsor for the project. Dr. Lee is an assistant professor and analytical chemist at Northern Arizona University researching organic electronics and photovoltaic cells. A TPD-SIMS device will help Dr. Lee and his laboratory gain the ability to analyze different materials ions. Sandia is interested in this project, because one of their devices is working inefficiently running because chemicals are desorbing off their materials while running. Sandia wants to know what and at what temperature these chemicals are desorbing.

## 1.2 Project Description

The project provided by the Sandia National Laboratory is to design, fabricate, and install a new sample holder and ionization mechanism. The original project description provided by Dr. Lee is as follows [5]:

> Northern Arizona University (NAU) has a home-built time-of-flight secondary ion mass spectrometer (TOF-SIMS) which is uniquely available for custom modification of sample holders and ionization sources. This device and NAU expertise will allow rapid testing and evaluation of approaches for nanosecond mass spectrometric analysis prior to designing an entirely new system. This approach will save Sandia National Laboratory both time and resources.
>
> The proposed work includes:
>
> 1. Design, fabrication, installation, alignment and testing of both a temperature programmed desorption (TPD) sample holder and ionization mechanism compatible with the current NAU time-of-flight (TOF) mass spectrometer (MS) system.
> 2. Modification of the current chamber for use with TPD.
> 3. TPD characterization of a Sandia stainless steel sample by TOF-MS system for comparison

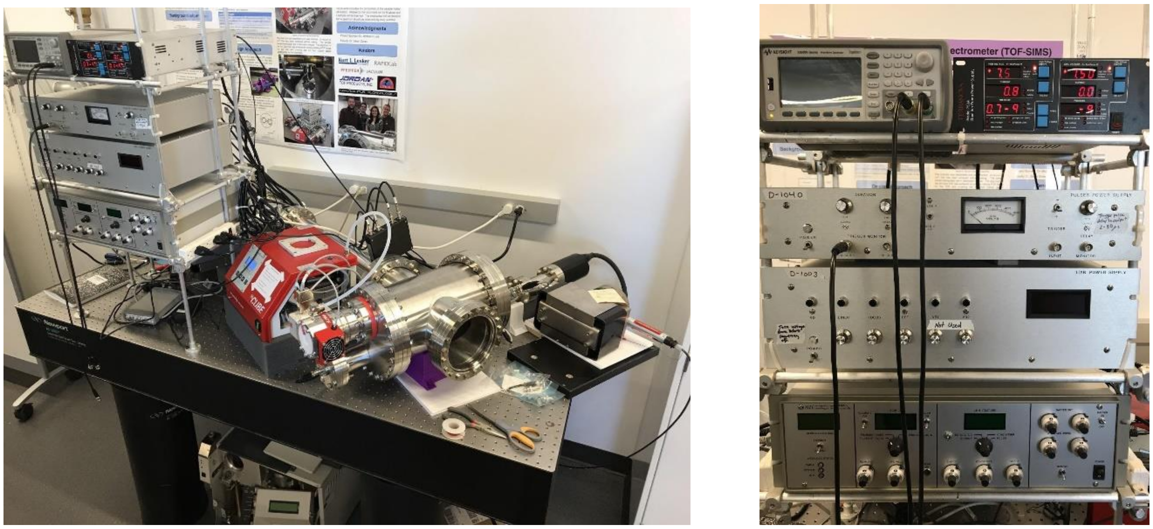
with Sandia data.

4. Report reviewing possibilities for ns resolution mass spectrometry

The sample holder must be re-designed to hold a larger, stainless-steel sample that could be compatible with TPD. The ionization mechanism must also be compatible with the current Northern Arizona University (NAU) time-of-flight (TOF) mass spectrometer system and with the new sample. It is required that the sample and the ionization source are aligned properly, and proper testing of the system has been performed. Overall, the main goal of this project is to modify the current TOF-SIMS chamber for use of TPD of a stainless-steel sample and compare with Sandia National Laboratory.

## *1.3 Original System*

The original system of this project it last year's TOF-SIMS capstone project. This TOF-SIMS includes a vacuum chamber, an ion gun, a sample holder with a ground attached, an ion funnel, a flight chamber, and a mass spectrometer. The setup of the original system can be seen in Figure 1.1 below.



(a) (b)

Figure 1.3: Current TOF-SIMS setup in the chemistry building. Figure (a) left depicts the vacuum chamber and pumps. Figure (b) photo shows the electronics' setup.

### 1.3.1 Original System Structure

The original system was made up of many different parts that were custom made for this machine. The main instruments include the two ion pumps (one 30L/s and one 60L/s), the turbo pump, the stainless-steel vacuum chamber, the ion gun that discharges cesium ions at the sample, the sample holder, and the mass spectrometer. which came out of the top the main chamber. The entire system sits on top of a two-ton table to dampen any vibrations on the TOF-SIMS system. It is located in a Dr. Lee's lab in the Chemistry building.

### 1.3.2 Original System Operation

When the project first began, the TOF-SIMS system was not operational. This was due to the ion guns power cable being damaged and the inability to get it fixed. The ion gun itself was operational but needed to be cleaned with joule heating. However, the turbo pump preformed perfectly, along with the ion pump. The vacuum chamber held the 10-7 torr pressure to be considered as a UHV. Even though the original design was not required to meet this high pressure.

### 1.3.3 Original System Performance

The TOF-SIMS system did not operate properly at the time this project began. The ion gun did not perform properly because a power cable connected to the ion gun was damaged. The cause of this damage is unknown to Dr. Lee. The cord was soon fixed by this capstone team by replacing the old cord. Once the cord was fixed, the system began to run again and was tested under orders of Dr. Lee to see how the ion gun would perform. However, this data was not saved onto the computer at that time; the system was merely turned on to see if it was working properly.

### 1.3.4 Original System Deficiencies

Last year's TOF-SIMS system does not meet any current customer requirements. It cannot heat a sample at a constant rate, it cannot support the new sample within the sample holder, and the ion gun cannot ionize the surface of the new sample. Also, TPD is not integrated into the original TOF-SIMS in any way. However, the original system can make the chamber an ultra-high vacuum, and the funnel can collect the activated ions. Overall, many modifications are necessary for the system to meet the current customer requirements.

# 2 REQUIREMENTS

The requirements for this project were given to by the faculty advisor, Dr. Michael Vernon Lee. These requirements will act as a guide in designing and building a sample holder that will allow for temperature programmed desorption (TPD) within the ultra-high vacuum chamber.

## 2.1 Customer Requirements (CRs)

To be able to develop a TPD SIMS device to the specifications of Dr. Lee, the team were given requirements that the design needed to meet. These requirements were received from the client and based on the project specified in section 1.1. From the project objectives, the following eight requirements were developed based on the client's preference and importance. Each requirement approaches the project objectives clearly and precisely and will help the team create innovating designs that the clients requested.

The customer requirements (CR) for the project are as followed:

1. The sample must be heated to a high temperature.
2. The sample must be heated at a constant rate.
3. There needs to be a system to measure the desorption data.
4. The TOF-SIMS system must be able to monitor the mass.
5. The turbo pump must be shielded from potential secondary ions within the chamber.
6. A new, functional sample holder must be designed and built to hold the sample in alignment to the extracting cone.
7. The entire system must be safe to handle and operate at high temperatures.
8. The cost of the design must not exceed the given budget.

To conduct the surface analysis experiment using TPD, the sample being tested must be heated to a high enough temperature to release the various particles within the surface. To analyze the TPD-SIMS data accurately, the sample must also be heated at a constant rate. The temperatures at which certain particles release from the surface, need to be known in order to reproduce the experiment. A thermocouple will be attached to the sample to measure the temperature and a mass spectrometer will read the mass per charge of the desorbed particles that have been ionized. If there is no way to measure the data, then there are no conclusive results in this experiment, that is why customer requirements one through four are necessary. The next customer requirement is to shield the turbo pump from ion gases. During experimentation, some particles will be released from the sample, but may not have been ionized. This creates a cloud of ions within the chamber with potential to damage the turbo pump. Moreover, the sample holder must be redesigned to meet the new customer requirements. There must be variability to the angle of the sample, the sample holder must withstand high temperatures, the sample holder must have reproducible positioning and alignment, the sample must also be removable from the system, and the sample holder must be electrically and thermally insulated. The electrical and thermal insulation will help make the system safe to handle and operate for anyone who may come in contact with the system. Lastly, the entire design must not exceed the total budget.

## 2.2 Engineering Requirements (ERs)

The project will provide Dr. Lee with a device that meets all the project requirements and constraints. The engineering requirements are specifications that give the customer requirements quantitative and qualitative goals. The engineering requirements are made to best fit the customer requirements and project objectives. After analyzing the specified constraints set by Dr. Lee, the engineering requirements were determined.

Customer requirements will be met through the following engineering requirements:

1. The sample will be heated up to 1200 °C.
2. The sample will be heated at a constant rate of 1 °C/s.
3. The desorption data will be measured with a time resolution of 2 nanoseconds.
4. The system should monitor mass of 50 mass per charge or lower.
5. The turbo pump must be shielded from at least 90% of the ion gas in the chamber.
6. The sample must be aligned with a tolerance of 10 nm.
7. In order to keep the system safe to users, the outer chamber must not heat to temperatures higher than 40 °C.
8. The thermal insulation must have a melting temperature greater than 1200 °C to keep the system safe and insulated.
9. The electrical insulation needs to have a resistivity of at least 106 Ω-cm to keep the electricity from reaching the chamber.
10. The design cost must be under $41,000.

## 2.3 Testing Procedures (TPs)

To conclude the effectiveness of the team's design and achieve the engineering requirements, measurements need to be taken to evaluate the designs worth. In order to accomplish these measurements, procedures were constructed. These procedures may be similar to the engineering requirements and may seem overly simplistic. The first few procedures entail checking the dimensions of the sample holder. Each piece needs to fit within the chamber perfectly in order to have the sample holder be removable. The following test also need to be completed; check security, adjustability, and alignment of the sample when in the holder, as well as test to ensure the entire system is secure. The next test procedure consists of measuring the temperature and desorption of the sample. This will be done through analyzing data from the thermocouple and the SIMS. Another testing procedure consists of checking the vacuum pressure within the chamber is about 10-7 torr. Once the overall system test is done, the team can analyze the turbo pump to determine the correct action to take, to shield it from ion gas. Then the team can do small tests to determine the effectiveness of a shield. Lastly there is a procedure to check all totaled parts cost in order to be below $41,000.

The following is a list of the procedures discussed:

1. Dimensioning of sample holder, wedge, and clamps
2. Check security, adjustability, and alignment of sample
3. Test the security of the entire system
4. Measure temperature and desorption
5. Check vacuum pressure
6. Monitor turbo pump during tests
7. Quick performance tests to determine if turbopump shielding is effective
8. Check part cost and total design cost frequently

## 2.4 House of Quality (HoQ)

The House of Quality (HoQ) is a diagram used for defining the relationship between customer requirements and the firm/product capabilities. The HoQ developed for this project can be found in Appendix A. The HoQ has led to the understanding of important customer and engineering requirements through comparison and reasoning. Each customer requirement was given a weight from one to ten, ten being the most important and one the lowest. Then each customer requirement was given a score to signify a collaboration to the engineering requirements. The score ranges from zero, one, three, or nine; where nine is an exact correlation. The engineering requirement of absolute technical importance (ATI) is that the insolation used must have a high melting temperature. This implies that the concepts developed must include this important aspect. Project cost and insulation resistivity were the next highest rated requirements in relative technical importance (RTI). The RTI score was based on the score received in ATI and provides the team with two more requirements that will be considered when developing designs. Overall the HoQ provides the team with a basic knowledge of the most important requirements to meet for this project.

# 3 EXISTING DESIGNS

TOF-SIMS systems have been around for over 20 years and have greatly developed in concept and in design [6]. Due to the developed of TOF-SIMS, many other systems have been created and are available for researching this project. By using multiple sources of research such as professors, scholarly articles, and physical interaction with last year's project, a lot has been learned about the TOF-SIMS systems.

## 3.1 Design Research

The design research done for this project has been done both online and in person. Much of our understanding of last year's capstone has come from physical interaction with the system itself. Through weekly meeting with Dr. Lee in the chemistry building, we have been able to experiment with and analyze the TOF-SIMS system. The locations of the ion gun, turbo pump, ion pump, flight chamber, and sample holder have been observed through this physical interaction. Personal interaction with Dr. Lee has been tremendously helpful in understanding the project. During the weekly meetings, he explained in detail how every piece of the system operates and what it does. He demonstrated this using drawings, examples, and references to the physical TOF-SIMS system.

Online research has also allowed us to gain a better understanding of the existing design. Each member was asked to research a particular topic in regards to the system. This allowed for all to share our individual findings and compile the research as a team. After background research on how each piece of the system works, team members used the internet to search for other existing ion guns, sample holders, and turbo pump shields.

## 3.2 System Level

The first molecular SIMS device was first invented in 1960 by Alfred Benninghoven in [8]. The TOF-SIMS used today was derived from the molecular SIMS. Time of flight SIMS systems are now available for consumer purchase. However, these devices are extremely expensive. Some existing designs include the TOF.SIMS 5 and the Hybrid SIMS.

### 3.2.1 Existing Design #1: TOF.SIMS 5

The TOF.SIMS 5 is a high-end instrument that is equipped to handle samples up to 300 mm in diameter. It has a 5-axis manipulator that allows for easy manipulation of the sample orientation [7]. The ion beam can also be manipulated in order to optimize the mass resolution. Other features of the TOF.SIMS 5 include heating the sample to a desired temperature, low noise vacuum system, and a 3D analysis of the sample [7]. Overall, this system is very advanced in the TOF-SIMS industry and has nearly all applications necessary for TOF experimentation.

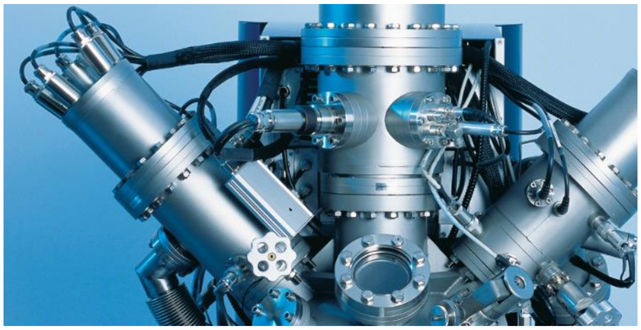


Figure 3.2.1: TOF.SIMS 5 vacuum chamber. [7]

### 3.2.2 Existing Design #2: Hybrid SIMS

The Hybrid SIMS is similar to the TOF.SIMS 5. It is an expensive, high-end SIMS instrument, used to analyze the composition of the secondary molecules within a material. However, the Hybrid SIMS can analyze organic and inorganic samples, while the TOF.SIMS 5 cannot. Also, it has an extremely high mass resolution of 240,000 at 200 m/z and a mass accuracy of less than 1 ppm (parts per million) [7].

## *3.3 Functional Decomposition*

Analyzing the functional decomposition of a system helps in understanding how the system functions. A black box model gives an overall understanding of the inputs and outputs of the system. Moreover, a functional model helps in understanding how each subsystem is connected and how each subsystem functions in the overall system. With these functional decomposition analytical methods, successful designs can be developed.

### 3.3.1 Black Box Model

The Black Box model is an evaluation used to determine the inputs and outputs of a device without knowledge of the internal workings. The model requires energy, mass, and sensor input and outputs. Figure 3.3.1, shows the Black Box model for the TPD SIMS device. The energy inputs consist of initial human and electrical interactions. The mass inputs include the steel sample and the electrons from the electron gun. The sensor input is the on/off process. The energy output corresponds with the inputs and produces an electrical output. The mass output is the product of secondary ions from the sample. The sensor output is similar to the input; the device uses an on/off process and produces noise. The primary function of the TPD SIMS device is to test dissertation data gathered in the ultra-high vacuum chamber.

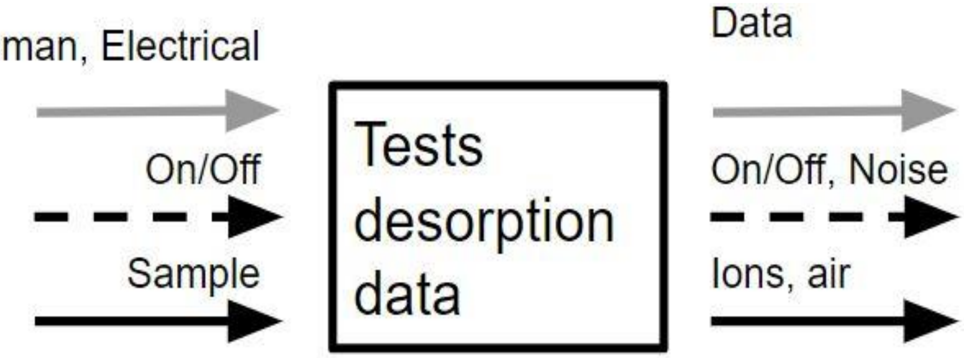


Figure 3.3.1: Black box model

### 3.3.2 Functional Model

Within this project, the team is redesigning a sample holder, updating an electron gun, and adapting a turbo pump shield within the vacuum chamber. A functional decomposition model is a structured representation of the functions within the modeled system. All components will need to be functioning together with the new additions, so it is important to understand each component and all of its functions within the device. The parts that will be purchased include the electron gun and a possible turbo pump shield. The two pumps attached to the vacuum chamber, computer/computer program, and TOF SIMS are already in the team's possessions thanks to the previous team's work. Figure 3.3.2 show the functional model for the TPD SIMS with the updated electron gun. It is important to fully understand the device and all the components within it in order to develop solutions to the improvements needed to be made.

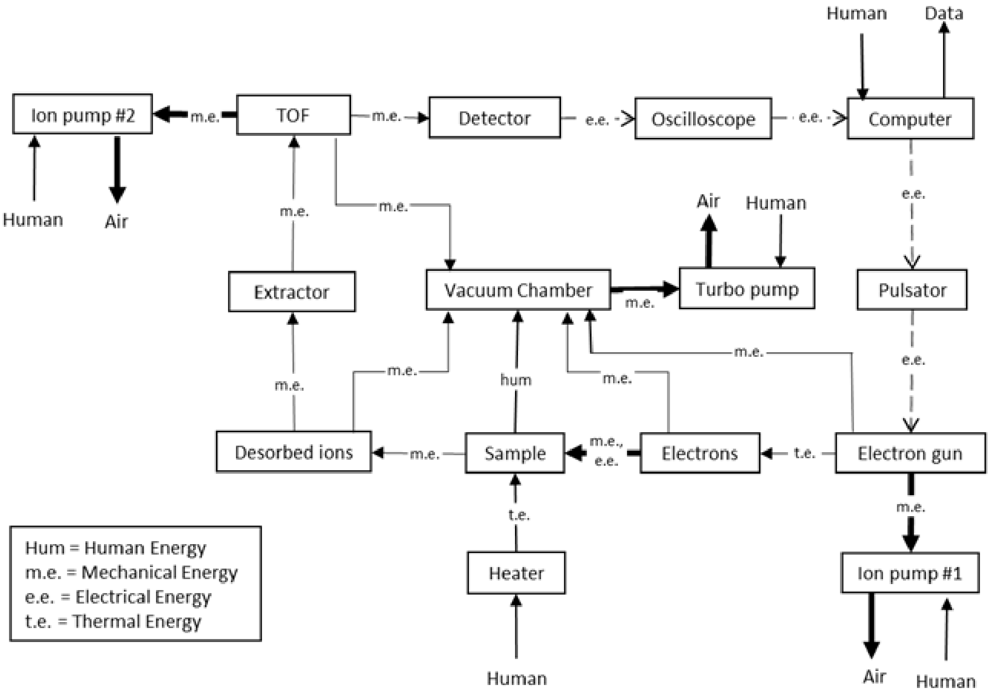


Figure 3.3.2: Functional model

## 3.4 Subsystem Level

Subsystems of the overall TOF-SIMS system were identified using the functional model combined with the customer needs. Defining these subsystems helped to identify individual parts that need improvement. The three main subsystems that will be analyzed include the sample holder, the source of ionization, and shielding of the turbo pump. Through online research, various existing designs for each subsystem were found and studied.

### 3.4.1 Subsystem #1: Sample Holder

The sample holder currently being used in the TOF-SIMS is a long rectangular piece of steel. At the end of the steel plate, the actual sample holder, a small receptacle, holds the material at a 45-degree angle using a spring to hold it steady and in place. This allows the ions that discharge out of the ion gun to hit the sample and bounce into the flight chamber. This entire system pulls out linearly from the left side of the chamber. A goal of our project is to redesign and create a sample holder that can withstand high temperatures and is electrically insulated. Because our sample with be electrically heated, the sample holder must be properly insulated in order to keep the user safe from any harm. If the holder is not insulated, the chamber would be subjected to electricity and thermal activity. The new sample holder must hold a sample of a larger size and different material from what was previously used. The new design should not permanently hold one specific sample, but must be interchangeable. This subsystem is important to the overall project because the sample needs to be held steady and aligned perfectly with the ion gun in order for the machine to work properly.

#### 3.4.1.1 Existing Design #1: Translucent Sampling Fixture

One example of a sample holder is a translucent sampling fixture. This sample holder allows for linear adjustment in relation to the placement of the sample and can be seen in Figure 3.4.1. It is manually adjusted and gives a ruler at the bottom for reference. This type of sample holder is mainly used to measure translucence of oddly shaped samples [9]. This design is helpful for our project with respect to its linear movement.

Figure 3.4.1: Translucent Sampling Fixture [9]

#### 3.4.1.2 Existing Design #2: Variable Angle Transmission Holder

Another example of an existing sample holder is the variable angle transmission holder and can be seen in Figure 3.4.2. This device can hold various samples sizes at various angles. The angle changes with human interaction twisting the sample mounting stage. This device is mainly used to measure transmission [10]. It could be used in this experiment by allowing us to accurately angle the sample at the ion gun while still being able to move it around when needed.

Figure 3.4.2: Variable Angle Transmission holder [10]

#### 3.4.1.3 *Existing Design #3: Multi-Purpose Specimen Mount*

Lastly, the multi-purpose specimen mount can hold many different shapes of specimen. This includes irregular shaped or perfectly cylindrical specimen up to one inch in diameter as seen in Figure 3.4.3 [11]. This may be useful in designing the new sample holder because it can hold many different material shapes non-permanently.

Figure 3.4.3: Multi-Purpose Specimen Mount [11]

### 3.4.2 Subsystem #2: Ionization Mechanism

An ionization mechanism refers to an instrument that generates a beam of heavy ions with a well-defined energy distribution. The beam is produced from plasma confined within a volume, and the ions of the energy are extracted and accelerated. The original TOF-SIMS project used an ion gun that discharged Cesium ions. However, due to the change in sample material, these ions are too large. The team needs to research other alternatives in ionization for the new project. Alternate ionization instruments include flood guns, a smaller ion gun, or an electron gun. This source must work at least at 70 eV, 50 µA and have an ion beam diameter of about 5mm. This will allow for a more efficient ionization operation for our new material of steel. All while staying within budget, specifically under $10,000.

#### 3.4.2.1 *Existing Design #1: Flood Gun FS 100*

A flood gun is an electromechanical device that provides a steady flow of low energy electrons to a desired target. Figure 3.4.4 shows Flood gun FS 100; this flood gun is distributed by OmiVac, a science equipment developer and supplier. This flood gun has a range of 0 to 500 eV, a max beam diameter of 11 mm, and a range of 0.1-1500 μA. This flood source is used to neutralize positively charged samples in SIMS systems [12]. This could be a potential source used in the project because it meets all the requirements as stated earlier.

Figure 3.4.4: Flood Gun FS 100 [12]

### 3.4.2.2 Existing Design #2: Ion Gun Package

This existing design includes an ion gun and an electronic control unit and can be seen in Figure 3.4.5. It can operate at voltage as low as 100 eV, with a 10mm beam diameter, and has a range of 1 to 18 μA. This ion gun is UHV compatible and is used to remove surface material [13]. This source also falls in the range of requirements and may be used in this project.

Figure 3.4.5: Ion Gun Package [13]

### 3.4.2.3 Existing Design #3: Model: ELS100

This last ionization source is very similar to the ion gun package. However, this instrument is an electron gun rather than an ion gun. This electron gun can be seen in Figure 3.4.6. This gun is mainly used in energy loss spectroscopy. It has a range of 5 to 100 eV, a 1mm beam diameter, and is UHV compatible [14]. Given its specification, this source may be of potential use for our project.

Figure 3.4.6: Electron Source ELS100 [14]

### 3.4.3 Subsystem #3: Shielding Apparatus

The turbo pump is located above the current sample insertion plate. Its main role is to help the ion pump in dropping the pressure when the pressure within the vacuum is relatively high. Since the turbo pump is attached directly to the main chamber, the turbo pump within the TOF-SIMS system is prone to being destroyed if any ions were to hit its blades. This means that the turbo pump cannot interact with stray ions and must be shielded. Currently, there is no turbo pump shield apparatus. It is not known whether or not this is needed for our project. Extensive testing must be done prior to understand its necessity. There may or may not be ion gas build-up within the TOF-SIMS chamber. This shield, if needed, will be located in front of the turbo pump intake pipe at an angle that will keep out any ion gas, yet will not totally cover the entrance.

#### *3.4.3.1 Existing Design #1: Magnetic Shield*

A magnetic shield is used to reduce in a space by blocking the particles with a barrier made of conductive or magnetic material. This design uses a magnetic field to shield a turbo molecular pump from plasma gas. This plasma gas may be produced by the electron gun interacting with the sample. This magnetic shield. However, it is still in experimental stages and is not durable, yet it seems to work well [15].

#### *3.4.3.2 Existing Design #2: Splinter Shield*

Splinter shields consists of a cone structure made out of a durable material that can withstand UHV pressure and protect the pump from ions. The Splinter shield device is shown in Figure 3.4.7. This shield is mainly used to protect a pump from large, coarse objects. It collects these foreign objects and keeps them from entering the suction chamber. Due to its design, it will cause a loss in efficiency of the pump [16].

Figure 3.4.7: Splinter Shield [16]

#### 3.4.3.3 Existing Design #3: MicroMesh Screens

Lastly, a micromesh screen may be used to protect the turbo pump from large ions, while also letting small secondary ions through. It is similar to any other screen as it has small square holes, but on the scale of 8 microns. It has a smooth surface that is easy to clean. Many micro mesh screens can be made out of gold, nickel, and copper. This type of shield has many applications including, electron ion separation, mass spectrometry, and nuclear particle sorting [17].

# 4 DESIGNS CONSIDERED

Understanding and utilizing the past device through a functional model allowed the team to develop ten concept designs that help the system meet the customer requirements. Each design takes a close look at the potential subsystems within the TPD SIMS. The team gathered to brainstorm and develop each design in order to produce innovative and creative outcomes that still meet the project objectives.

## *4.1 Sample Holder Designs*

The following sections of this report will describe in detail various conceptual ideas for the sample holder. Five designs in total were conceptualized and drawn out. Included are figures with the concept drawings and tabulated pros and cons for each design.

### 4.1.1 Design #1: Insulated sample holder coming in from the side

This design, shown in Figure 4.1.1, insulates the chamber from heat and electricity from the sample. The secondary arm that holds the sample is insulated thermally from the sample by a material with a high heat resistance and that can withstand a temperature of 1500 °C. The primary arm is insulated electrically from the secondary arm by electrically insulated washers between the two arms and the bolt. The bolt is also surrounded by a sleeve so that electricity cannot run through it. The design pros and cons can be found in Table 4.1.1.

Table 4.1.1: Pros and Cons for Design 1

| Pros | Cons |
|---|---|
| Could reuse same secondary that is already holding the sample | Cantilever beam could cause high amounts of stress in the arms |
| No wasted energy while heating up the sample holder | Secondary arm has an electrical potential |
| Holds sample stationary | Would be difficult to manufacture electrically insulated sleeve |

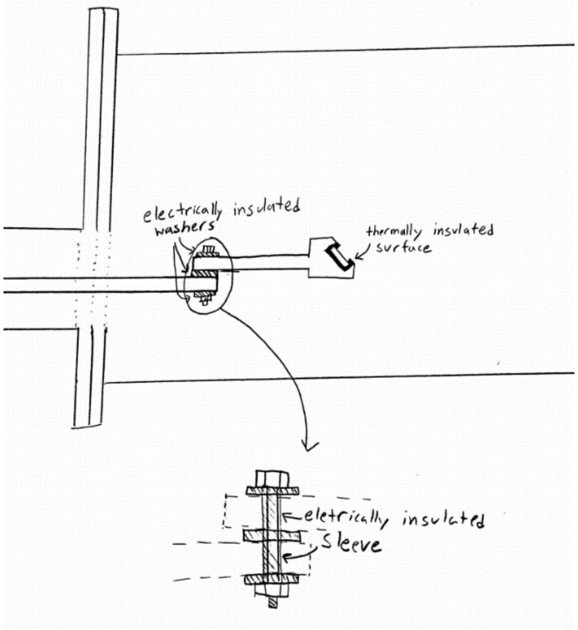


Figure 4.1.1: Insulated sample holder coming in from the side

### 4.1.2 Design #2: Carousel sample holder

This sample holder has four samples on the holder with each of them thermally insulated and the motor rod is electrically insulated. When the data for one sample is collect the motor is turned until the next sample is in place and then the next test is done. The design can be located in Appendix B, Figure B1. The list of pros and cons can be found in Table 4.1.2.

Table 4.1.2: Pros and Cons for Design 2

| Pros | Cons |
|---|---|
| Multiple samples remain inside vacuum | Difficult to heat up each sample individually |
| No human interaction with inside of chamber required, until a sample is not available | More weight on cantilever beam |
| Can be angled at 45$^o$ easily | More energy sources into the vacuum |
| Not bulky and could reduce costs | Difficult to align very precisely |

### 4.1.3 Design #3: Hanging sample holder with wires being the heating source

This sample holder design, shown in Figure 4.1.3, is held at the top of the chamber with a wire that will be used for joule heating the sample with the holder thermally insulated with a material that has a low electrical resistance. To implement this design, a hole would have to be drilled into the top of the chamber and then sealed again. Drilling then sealing the chamber would be both costly and time-consuming. Also, the ion cone is directly above the sample making inserting the sample harder. The pros and cons list can be found in Table 4.1.3 below.

Table 4.1.3: Pros and Cons of hanging sample design.

| Pros | Cons |
|---|---|
| Sample holder doubles as the heat source | Costly |
| No need for electrical insulation | Not very stable within chamber, variation in sample location, not very precise |
| Very minimal design, not a lot of bulk or materials | Chamber will need to be modified to facilitate |
| Ease of attaching sample to holder | Will be difficult to orient sample and keep at 45° |

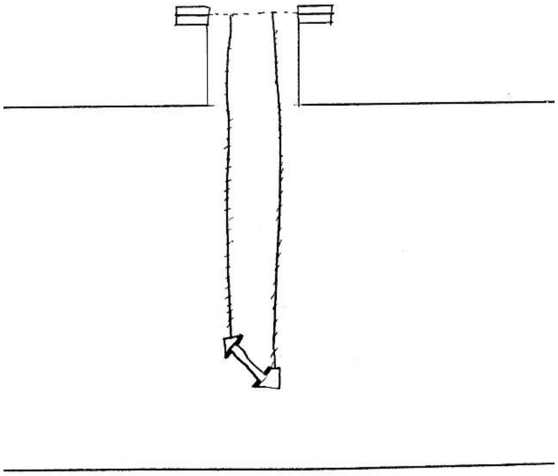


Figure 4.1.3: Hanging sample holder

### 4.1.4 Design #4: Remote controlled robot with rack of extra samples

This sample holder is a robot that is remotely controlled. It heats the sample by using a lazar and it can also grab other samples and place them into the chamber before running the experiments. The pros and cons are listed in Table 4.1.4 and a picture of the design is in Appendix B, Figure B2.

Table 4.1.4: Pros and Cons of remote controlled robot and a rack of samples within the chamber.

| Pros | Cons |
|---|---|
| Multiple samples without breaking the vacuum | Very expensive |
| Robot is thermally insulated | The sample is subject to a lot of movement, variation in placement |

| | |
|---|---|
| One cord connected to robot | Chamber is curved, could be difficult to control robot |
| Holds the sample stationary while in position | Samples on rack may heat up during testing and produce gases |

### 4.1.5 Design #5. Sample Introduction Chamber System

This design features an introductory vacuum chamber that has a gate between the introductory chamber and the testing chamber. First, the introductory chamber is brought to a vacuum. Next, the gate is opened from the main chamber into the introductory chamber, and the sample is extracted into the introductory chamber. After this, the gate is closed and the vacuum in the introductory chamber is relieved so the sample can be changed. After the sample is changed, the introductory chamber is brought to a vacuum, the gate is opened, the sample is put in the main chamber, and the gate is closed again. For this design, a secondary pump will be needed as well as a magnet that will control the sample holder while inside the chamber. This design is pictured below in Figure 4.1.5 and is based off similar designs used at Sandia National Laboratories and on the TOF.SIMS5 commercial setup [7]. The pros and cons of this design are also summarized in Table 4.1.5.

Table 4.1.5*:* Pros and Cons of a sample introduction chamber system.

| Pros | Cons |
|---|---|
| Never have to break the vacuum in the testing chamber | Very expensive |
| Easily change sample | A lot of materials, bulky and has space requirements |
| Degassing time decreased | Magnetic control may interfere with experiment and turbo pumps |

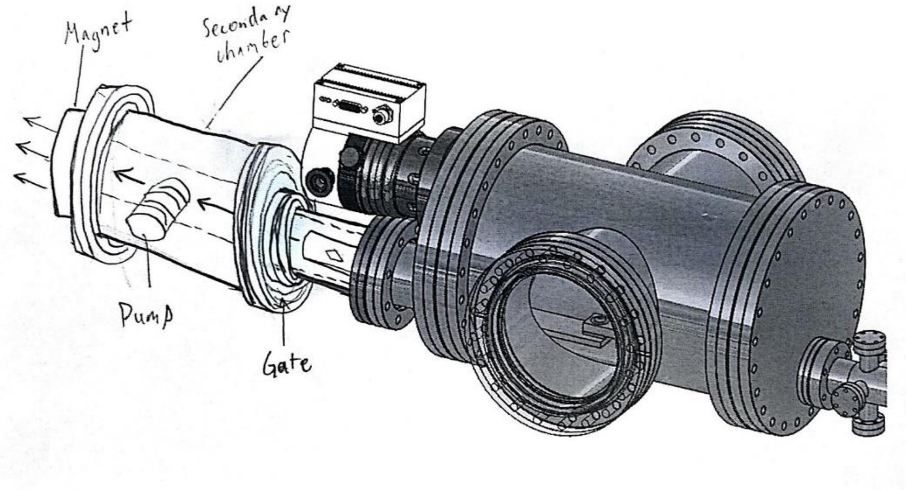


Figure 4.1.5: Sample Introduction Chamber system.

## 4.2 Turbo Pump Shielding Designs

### 4.2.1 Design #6: Disconnecting the turbo pump while system is running

This design cuts the turbo pump off of the vacuum with a vacuum gate. This allows the pump to be turned off. With it being disconnected the chamber can be dropped to a lower pressure, since it is thought that the turbo pump is the limiter on the pressure. The pros and cons have been listed in Table 4.2.1 and a visual representation in Figure B3 in Appendix B.

Table 4.2.1: Pros and Cons of using a pressure gate to shield turbo pump from shock.

| Pros | Cons |
|---|---|
| Turbo pump may be turned off during testing | Ion pumps have to pull all the secondary ions out |
| When turbo pump is disconnected, the chamber can reach a lower pressure | Pressure gates are expensive |

### 4.2.2 Design #7: Splinter Shield

The design in Figure 4.2.1 utilizes a splinter shield designed specifically for this turbo pump to shield the pump from shock due to foreign particles. The manufacturer of the turbo pump company, Pfeiffer Vacuum, produces a splinter shield for this pump that would work well. [16]. This design is pictured in Figure 4.21 below. A table with the main pros and cons is also below in Table 4.2.2.

Table 4.2.2: Pros and Cons of splinter shield design to shield the turbo pump from shock.

| Pros | Cons |
|---|---|
| Turbo pump is constantly shielded under vacuum | Reduction in pumping speeds due to poor conductance |
| No accumulation of particles or debris in suction chamber | Lifecycle could be reduced if large particles get caught in shield |

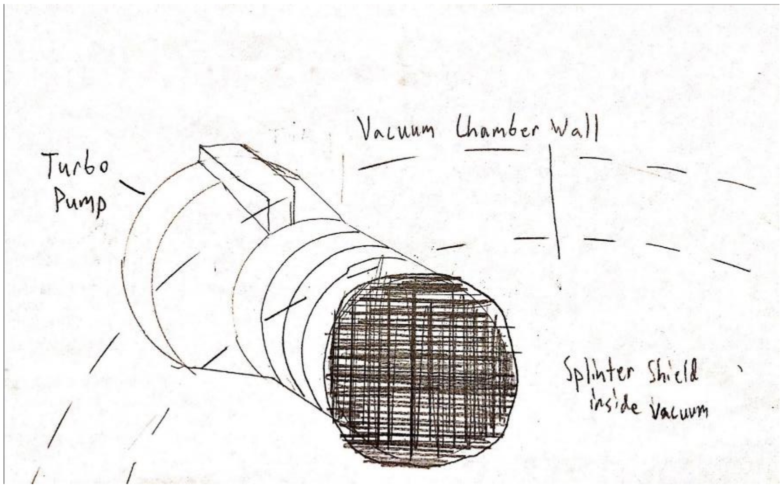


Figure 4.2.1. Turbo pump shielded from shock using a splinter shield

### 4.2.3 Design #8: Curved entry into the turbo pump

In this design the pipe leading into the turbo pump is turned upward. The curved pipe would make any atoms that get into the turbo pump lose most of its energy before reaching the pump. The design can be located in Appendix B, Figure B4. A pros and cons list can be seen in Table 4.2.3.

Table 4.2.3: Pros and Cons of curved entry into the turbo pump design

| Pros | Cons |
|---|---|
| Easy to implement | May not be completely protected |
| Inexpensive | |
| Does not restrict flow | |

## *4.3 Electron Gun Designs*

### 4.3.1 Design #9: Electron Gun

An electron gun is used to accelerate electrons off a sample at a high velocity. In Section 3.4.2, existing flood guns and electron guns on the market were discussed and researched. The four vendors mentioned have been contacted and two have responded with prices. While it is possible to construct an electron gun from various components, for this experiment, Dr. Lee and the team have chosen to purchase an electron or flood gun. The reason behind this decision is designing and building an electron gun for use in TOF SIMS experiments to get precise measurements, is not in the scope of this project. Using a homemade electron gun can also be dangerous and would be difficult to produce, in order to meet the precise requirements detailed in Section 2.2. Therefore, the team has done research on the four options in Section 3.4.2 and will select one to purchase with Dr. Lee's approval based on the required beam current, beam diameter, and electron voltage.

## *4.4 Total Redesign of System Setup*

### 4.4.1 Design #10: Vertical TOF-SIMS Vacuum Chamber

In Figure 4.4.1, the existing system setup has been reoriented and remanufactured to have a vertical time of flight chamber. Since the chamber is at a low pressure creating a UHV of around 10-9 Torr, gravity will not affect a small particle in it's flight path. This information allows for the flight chamber to be redesigned with an upwards orientation. Overall, the design saves a physical space in the laboratory. This design is a potential design for the secondary phase of the project which is initializing the research and design phases of a nanosecond resolution temperature programmed desorption (TPD) TOF-SIMS vacuum chamber. This design also features the sample introduction chamber to easily change samples. The pros and cons list of this design can be seen in Table 4.4.1.

Table 4.4.1. Pros and Cons of a vertical TOF-SIMS setup

| Pros | Cons |
|---|---|
| Reduces physical space needed for test setup | Could become off balance |
| Utilizes an introduction chamber system | Very expensive (two turbo pumps, ion pumps, ion or electron gun, sample holder, etc.) |
| Two turbo pumps will accelerate pumping speed | Difficult to orient ion pumps in good positions |

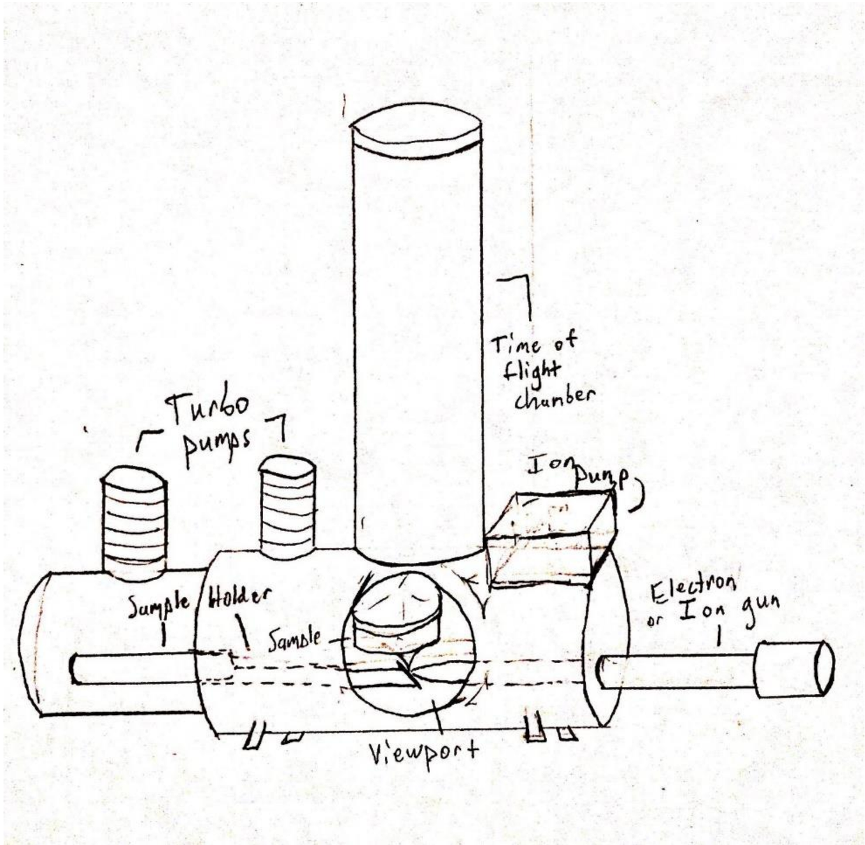


Figure 4.4.1. Vertical TOF-SIMS experiment setup

# 5 DESIGN SELECTED

This chapter will discuss the team's rationale for selecting a design in the near future. The methods utilized will be discussed to validate our decision on a final design.

## *5.1 Rationale for Design Selection*

As seen in the House of Quality in Appendix A, Figure A1, the most important engineering requirements are thermal and electrical insulation of sample holder, as well as lower cost of the overall project. These engineering requirements helped to develop numerous concepts which were later turned into considered designs. The considered designs described in section four were assessed in order to come to a final design. Each design was assessed on how well it met the customer needs and engineering requirements mentioned in section two. Dr. Lee provided insight and advice on selecting a final design that is capable of meeting all needs and requirements. The team did not need a concept design tool because multiple designs were constructed and consulted with Dr. Lee. With the help of Dr. Lee and his experience, the concept designs were narrowed down to determine the best designs for the project. Multiple CAD drawings were created of plausible designs in order see how each design would fit in the actual TOF-SIMS system. This method also helped narrow down possible design concepts. In the end, the final design was presented to the client in which the drawbacks, improvements, and experimental limitations were discussed. Once this was completed and all improvements were considered, the team finalized the CAD and prepared a list of materials for Dr. Lee to approve.

## *5.2 Design Description*

### 5.2.1 Electron Gun

The electron gun that has been selected to purchase will be the OmniVac Flood Gun FS100 that includes a PS-FS100 power source as discussed in Section 3.4.2.1 and pictured in Figure 3.4.4 [7]. The selection of this electron gun was made with the assistance of Dr. Michael Lee. The driving factor to purchase this specific flood gun was the low cost and the ability to customize the insertion length. The flood gun will be customized to have the correct insertion length for the current vacuum chamber and system. This modification to the insertion length will increase the overall cost of the flood gun. However, the overall price of the OmniVac flood gun is approximately $9,500, and is well within budget.

### 5.2.2 Thermocouple with Power Wires

A thermocouple is required to measure the temperature of the sample, and power wires are needed to heat the sample via a current. The Type K Mini Plug Plus Power Thermocouple from Kurt J. Lesker is capable of reading the temperature of a sample up to 1,250 oC. This temperature is above the temperature that the sample will be heated for this project. Moreover, this device is also capable of supplying a voltage of up to 12 kV and a current of 150 Amps [21]. The Kurt J. Lesker Type K Mini Plug Plus Power Thermocouple costs $443.00. This cost is also under the allotted $500 budget for the thermocouple power unit [21]. See Figure 5.2.2 below for the layout of this thermocouple. It can be seen from this figure that there are four wires connected to the device. Two of these wires are the power wires, while the other two are the thermocouple wires.

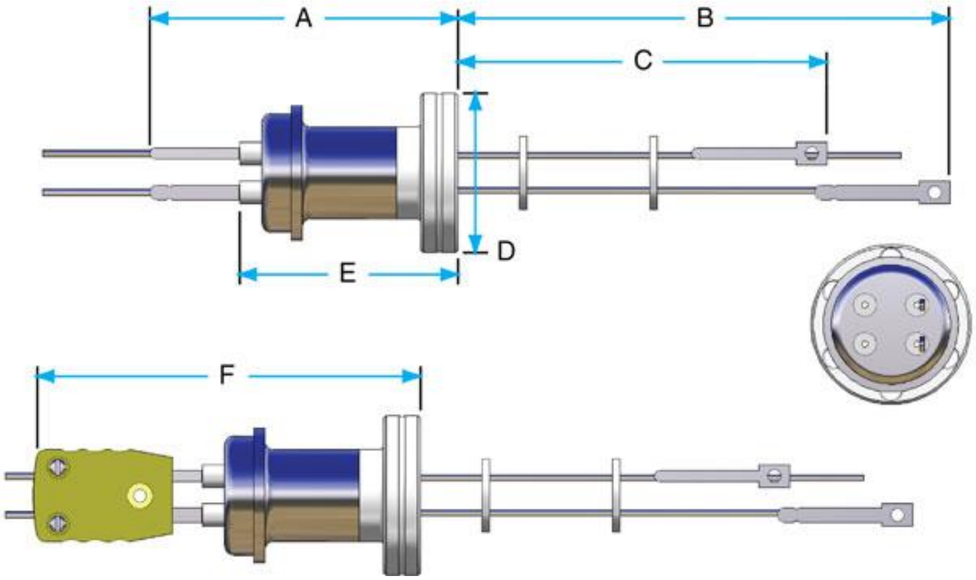


Figure 5.2.2: The Kurt J. Lesker Type K Mini Plug plus Power - CF Flanged, Single-ended thermocouple [18]

### 5.2.3 Sample Holder Design

The final concept design that has chosen for this project is pictured below in Figure 5.2.3.1. This design consists of two copper clamps that will hold the sample in place. The power wires from the thermocouple will be connected to the copper clamps. The copper clamps will conduct the electricity and heat into the steel sample. The thermocouple wires will be connected to the bottom of the sample in order to accurately measure the temperature of the sample as it heats up. Beneath the sample and the clamps is an Aluminum Oxide base insulator. This insulation will protect the overall system from being exposed to electrically current. Below the insulator is a wedge-like base. This base will be connected the bottom of the chamber using a small amount of vacuum safe adhesive.

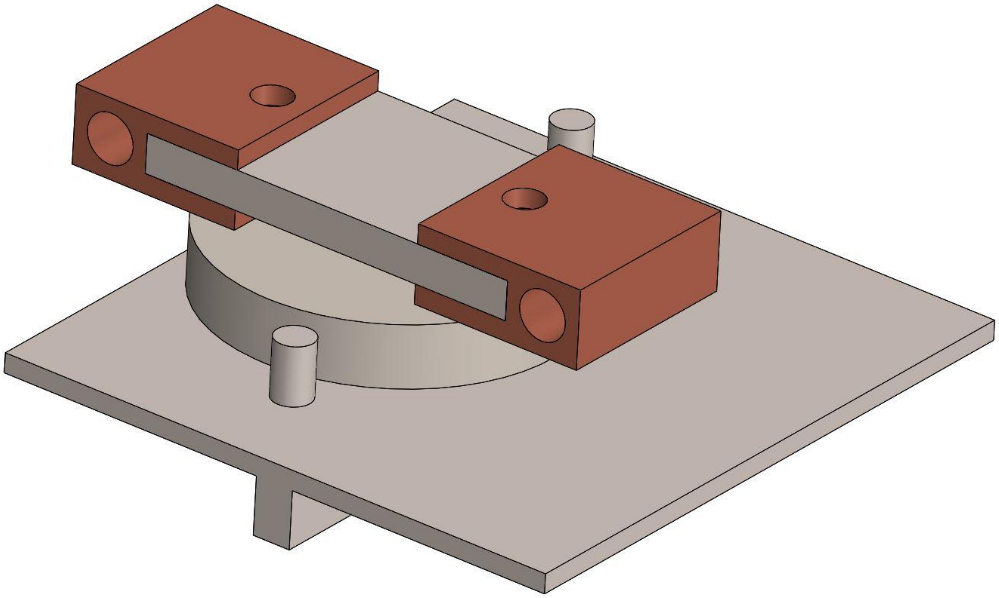
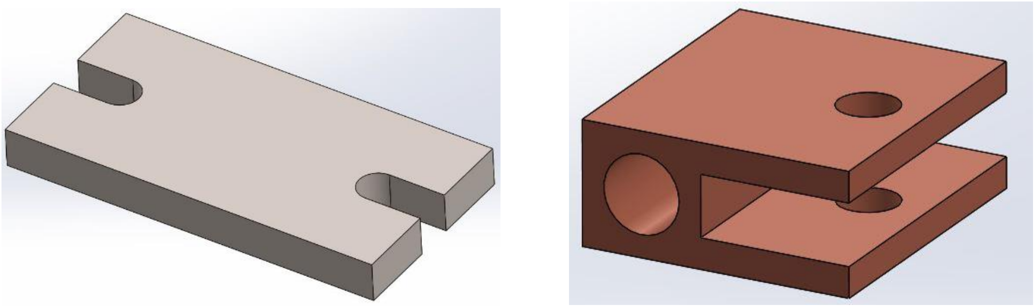


Figure 5.2.3.1: Final design assembly

Through the requirements provided above and client instructions, the team realized the sample would need to be designed with the intentions to be secured within the holder while also being removable. Figure 5.3.2.2a shows the newly designed sample that is needed to ensure productivity. The sample will be stainless steel, as indicated by the client, and have an exposed surface of 1 square inch. The sample was made with cutouts in order to be able to secure it with a bolt to the sample holder.

Through the requirements provided above and the client's instructions, it became apparent that the sample itself would need to be redesigned. To increase electrical efficiency, the copper plates should clamp down on the sample tightly. To do this, a screw will be tightened onto the copper clamps. This design can be seen in Figure 5.3.2.2(b). Therefore, the sample needs to be modified with cutouts to allow for this screw to tighten the copper clamps. Figure 5.3.2.2(a) shows the newly designed sample that is needed to ensure productivity. The sample will be stainless steel, as indicated by the client, and have an exposed surface between the copper plate of 1 square inch.



(a) (b)

Figure 5.3.2.2: The final design for the sample and clamp. Figure (a) depicts the sample design. Figure (b) shows the clamp design.

The sample holder base needs to be able to withstand high temperature, high electrical potential, and electron bombardment. Therefore, the base needs to be made of a material that is a good insulator such as alumina (Al3O2). Alumina was chosen based on a material analysis of various vacuum chamber allowable materials. For a material to be used in an ultra-high vacuum, it must have an outgassing rate below 10 -9 TorrL/sec [19]. Alumina is one of the few materials that is both an electrical and heat insulator and can be used in an ultra-high vacuum. This material can be injection molded which decreases time it takes to produce and reduces the cost. The two holes on the top of the sample holder base, seen in Figure 5.2.3.3(b), are where the clamps will be bolted to the base. These screw holes are not directly above the round divots pictured in Figure 5.2.3.3(a). These indents are used for alignment of the sample holder system. The three rounded divots will cover small ball bearings located on the wedge in order to secure the alignment of the sample.

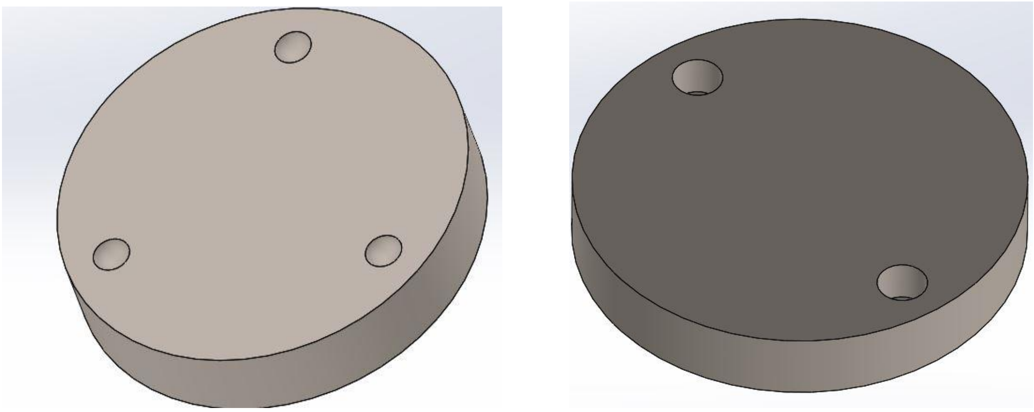

(a) (b)

Figure 5.2.3.3: Sample holder base bottom and top view respectively. Figure (a) Bottom view, (b) Top view

Even though the chamber is rounded, the sample holder must sit flat. The wedge design will allow the sample holder system to sit flat while the wedge holds it off the ground. Therefore, the wedge must be made from a strong material in order that the wedge does not bend under pressure. Thus, stainless steel will be used for the wedge because of its strength and low cost.  The middle leg under the wedge is rounded to the curvature of the bottom of the chamber. This allows for a more steady and stable wedge design. The round divots on the top of the wedge are for the ball bearings to be placed into for alignment of the sample holder base. This design can be seen in Figure 5.2.3.4.

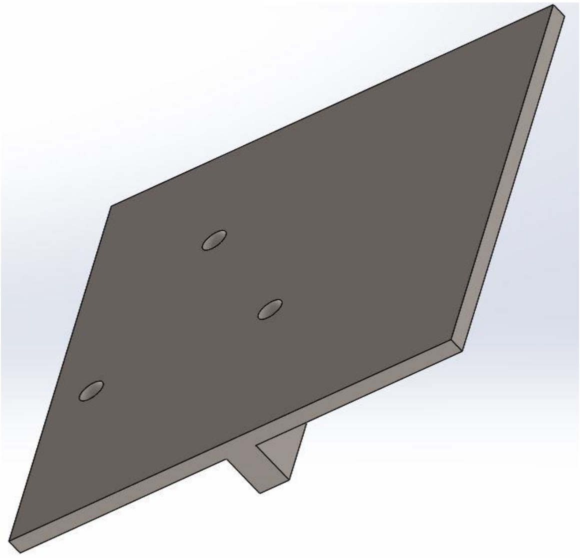


Figure 5.2.3.4: The wedge design

# 6 PROPOSED DESIGN – First Semester

The proposed design consists of a sample holder, a flood gun, and a thermocouple. Figure 6.1 shows the full sample holder system. This final design meets the all of the customer and engineering requirements as proposed above in section two. The sample holder design includes: two copper clamps that hold the sample in place, a cylindrical sample holder base, a wedge, three ball bearings, and two tightening screws. Table 6.1 lists the parts used, the number of parts, and the material it will be made of. However, Figure 6.1 and Table 6.1 only demonstrate the sample holder design the team developed themselves. It does not include the thermocouple and the flood gun. These devices will be items bought separately from the sample holder.

The materials needed for the sample holder will be bought from various industries. Once these parts have been delivered, the sample holder will be built according to the design shown in Figure 6.1. After the sample holder is completely finished then the team will begin experimenting with the system to test its performance.

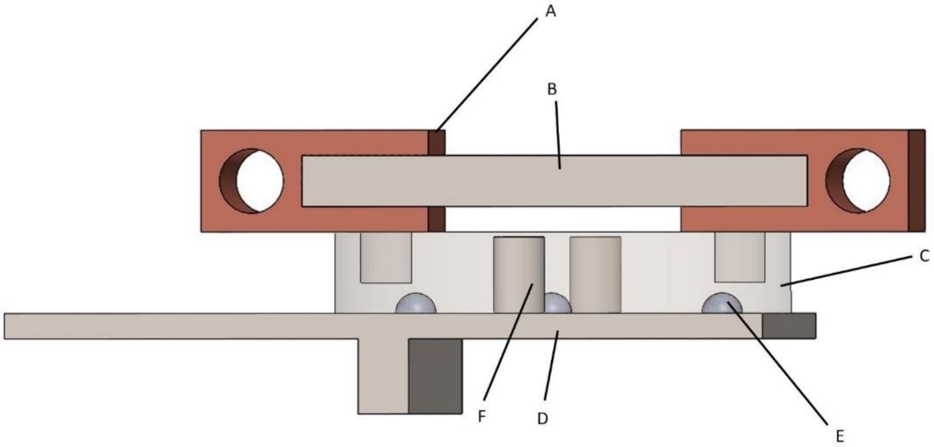


Figure 6.1: Sample holder design with parts alphabetically labeled

Table 6.1: Proposed Sample holder designs part list

| Part Label | Part Name | Number of Parts | Material |
|---|---|---|---|
| A | Copper Clamps | 2 | Copper |
| B | Sample | 1 | Stainless Steel |
| C | Sample Holder Base | 1 | Alumina |
| D | Wedge | 1 | Stainless Steel |
| E | Ball Bearings | 3 | Stainless Steel |
| F | Tightening Screw | 2 | Stainless Steel |

A bill of materials for this capstone project at this time is not appropriate. This is due to the nature of the parts being designed. There is little information on the cost of labor for various parts of the design. For example, the Aluminum Oxide sample holder base is a delicate part and needs special accommodations to produce the correct design. This part will have to be molded to the exact design specifications, since Aluminum Oxide is a ceramic and in the form of a powder which is too dangerous to cast in an uncontrolled environment. Another example is the sample itself, not only does it have to be machined to accommodate the sample holder, it also needs to be machined in a secluded environment with little to no outside contact. Since the bill of materials is not appropriate at this stage of the project, the team has included a tentative budget with anticipated costs as well as the source of the product in Table 6.2.

Table 6.2: Tentative budget

| | **Available** | **Anticipated** | **Sourcing** |
|---|---|---|---|
| Electron Gun | $12,000 | $9,500 | OmniVac |
| Thermocouple | $550 | $433 | Kurt J. Lesker |
| Power Supply | $4,000 | $2,000 | OmniVac |
| Gate Valve | $4,000 | $2,235 | NA |
| Sample holder | $5,000 | NA | NA |
| Travel Costs | $5,000 | NA | NA |
| Software | $800 | NA | SIMION 8.1 |
| Computer w/ DSP board | $2,000 | NA | Dell |
| Miscellaneous | $7,500 | NA | NA |
| **Total** | **$40,850** | **$14,168** | |

The table outlines the tentative budget the team was given by Dr. Lee. Each part and or category has an available price, which is the maximum amount of money allowed to be spent. The next section of the table is the anticipated price, which is the amount of money the team expects to pay for the part or category. Most have an NA due to the fact the team has not decided on where the part will be bought or how the available amount will be spent. The OmniVac Flood Gun FS 100 is  shown in figure 6.2 as the electron gun. It needs to be ordered relatively soon due to its 10 to 12-week lead time. While the Kurt J. Lesker Type K Mini Plug plus Power CF Flanged, Single-ended thermocouple will have an assumed lead time of 5 weeks. Both of these devices will be ordered within a week of obtaining any of the funds provided by our sponsor. All other pieces will be assumed to have a 2 to 3-week lead time for manufacturing and processing. These other pieces will be ordered before December 15th to ensure the parts can be assembled by January 16th. The device will be completely set-up and ready to test by March 30th and it should be calibrated and any data should be collected by April 28th..

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# 8 APPENDICES

## 8.1 Appendix A: House of Quality

Table A1: HoQ - Fall 2017

| | | Engineering Requirement | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Customer Requirement** | **Weight** | Porject Cost ($) down | Time Resolution (nanosecond) down | Monitor Mass (m/z) down | Insulation resistivity (ohms) down | Insulation melting temp (degree C) up | Sample alignment tolerance (nm) down | Heat sample at a constant rate of 1 C/s | Sheild Turbo Pump (% efficiency) up | Outer Surface Temp of Chamber (C) Down |
| Reliability | 9 | 9 | 3 | | | | 9 | 1 | 3 | |
| Durability | 7 | 9 | | 3 | 3 | 9 | | | 9 | 3 |
| Heat Sample | 10 | 3 | | | 3 | 1 | | 9 | | 1 |
| Heat Sample at a constant rate | 10 | 3 | | | | | | 9 | | 1 |
| Measure desoprtion time | 8 | | 9 | | | | 3 | | 1 | |
| Monitor mass | 8 | | | 9 | | | 3 | | 1 | |
| Shield Turbo Pump | 10 | 1 | | | | | | | 9 | |
| Design New Sample Holder | 10 | 9 | | | 9 | 9 | 9 | 1 | | |
| Protect TOF from high Temperatures | 9 | | | | 9 | 9 | | 3 | | |
| Purchase a new electron gun | 10 | | | | | | | | | |
| Safety | 10 | | | | 9 | 9 | | | 3 | 9 |
| Stay within Budget | 10 | 9 | 3 | | | | | 3 | | |
| Absolute Technical Importance (ATI) | | 323 | 129 | 93 | 312 | 334 | 219 | 256 | 226 | 131 |
| Relative Technical Importance (RTI) | | 2 | 8 | 9 | 3 | 1 | 6 | 4 | 5 | 7 |
| Target ER values | | $40,000 | 2 ns | 50 m/z | 106 ohms | 1200 C | 10 nm | 1 C/s | 90 % | 40 C |

## 8.2 Appendix B: Designs Considered

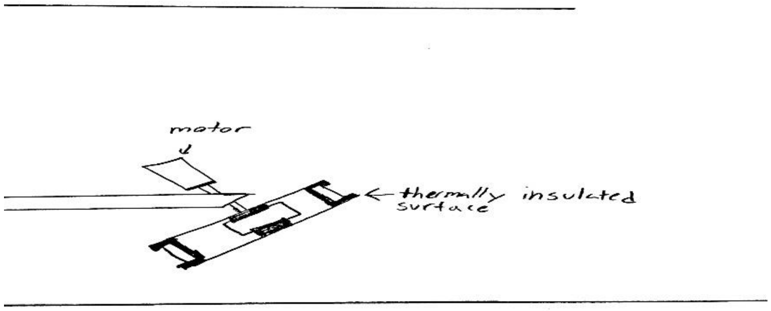

Figure B1: Carousel sample holder (design #2)

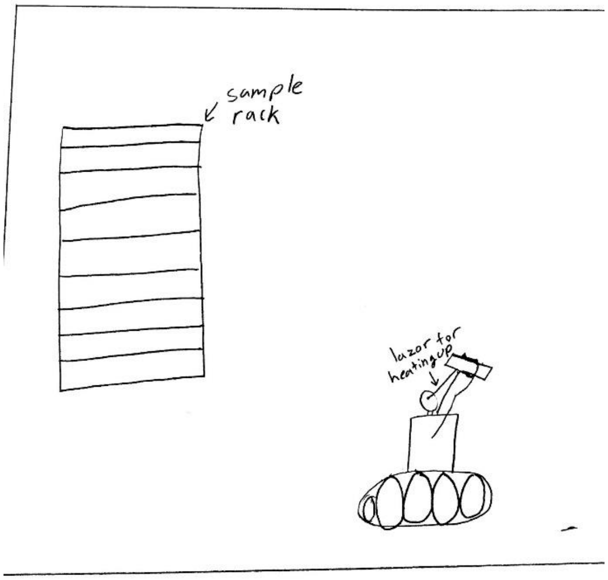

Figure B2: Remote controlled robot with rack of extra samples (design #4)

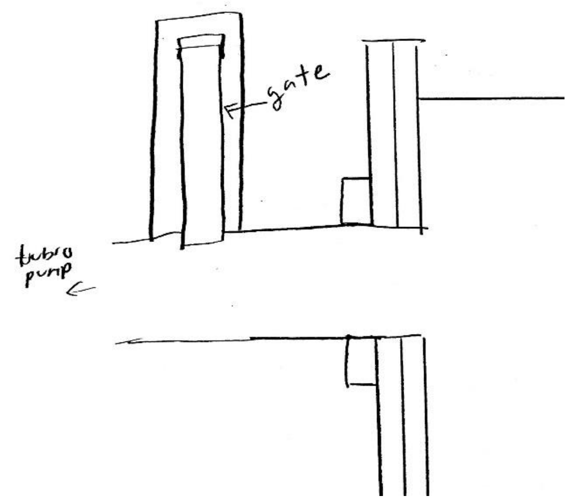

Figure B3: Disconnecting the turbo pump while system is running (design #6)

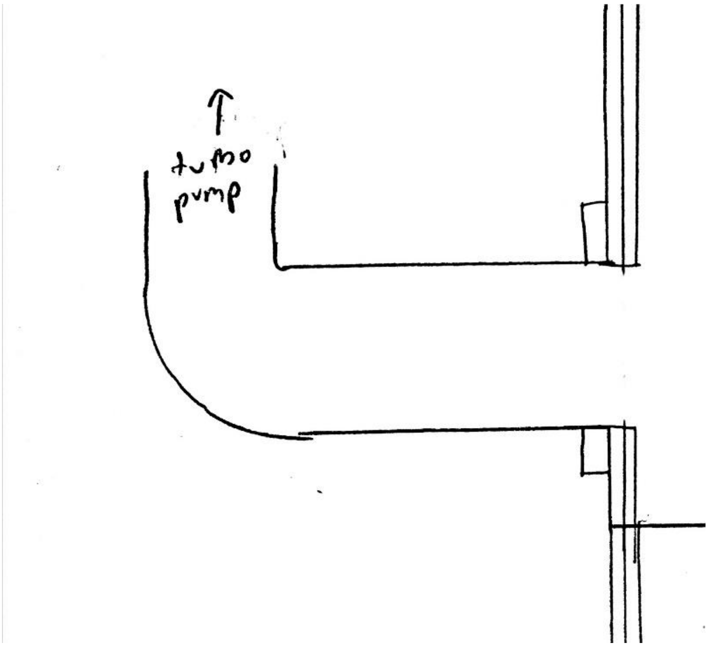

Figure B4: Curved entry into the turbo pump (design #8)