

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

Final Proposal

Nikki Cain

Colin Evans

Kirsten Larson

Dalton Stone

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**NORTHERN
ARIZONA
UNIVERSITY**

Project Sponsor: Sandia National Laboratory

Faculty Advisor: Dr. Michael V. Lee

Sponsor Mentor: Sean Simpson

Instructor: Dr. Sarah Oman

DISCLAIMER

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EXECUTIVE SUMMARY

The purpose of this project was to modify an existing time of flight (TOF) secondary ion mass spectrometry (SIMS) system with a vacuum chamber attached to it to measure temperature programmed desorption (TPD) data. A TOF-SIMS measures ions passing through it and send voltage data to a computer where they can be interpreted and stored as data. TDP is the process of a material outgassing certain molecules at a specific temperature. The sample that will be used in this experiment will be stainless steel and will be increased to a temperature of 1200 °C at a rate of 1 °C per second. The molecules will then will ionized by an electron flood gun, so the ions will be attracted to the positively charged entrance to the TOF-SIMS, also known as cone throughout the document. Heating up the sample was done with joule heating and the temperature was found using k-type thermocouples.

The design and testing of the sample holder was done by the team while other parts were purchased through outside manufacturers. The sample holder was made out of a stainless-steel wedge to allow the base to have a level surface to rest on. An alumina insulating base was added to the sample holder to reduce heat and current from reaching the through the chamber walls, as well as two copper clamps that was used to transfer power into the sample, heating it up. A securing system for holding the insulating base to the wedge and a silicon cantilever to hold the thermocouple wires to the bottom of the sample, was also added to the sample holder. Between the insulation and wedge, there will also be three ball bearings to make the location of the insulating base reproducible. Parts of the wedge was glued to the bottom of the vacuum chamber with ultra-high vacuum (UHV) compatible epoxy, to ensure stability. These components of the sample holder were designed to fit into the existing TOF-SIMS with low tolerances.

Through research of multiple companies, the flood gun was purchased from OmniVac for \$9500. The thermocouples and power feedthroughs were purchase as one from Kurt J. Lesker for \$433. The nuts and bolts were purchased from McMaster's. The other components were manufactured by other sources including the local machine shop on campus and the team themselves. After all parts have arrived, the team tested all parts, and upon approval, assembled and installed all components into the current system and begin to acquire TPD data measured from the TOF-SIMS system setup.

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1 BACKGROUND

1.1 Introduction

The goal of this project was to modify and enhance a Time of Flight Secondary Ion Mass Spectrometer (TOF-SIMS). A TOF-SIMS incorporates an ion source that generates ions, a collection cone which collects the particles, a time of flight chamber which transports the ions, and a mass spectrometer that measures the particles and provides the data [1]. This method of mass spectrometry is used by scientists and researchers to analyze the number of secondary ions removed from the surface of a sample [2]. It is also used to understand the 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 ion collides into the sample, 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 allowed the team to take the measurements needed to fully analyze a sample within seconds.

The project's objectives were; to keep the chamber at a vacuum state of about 10^{-7} torr, make a sample holder with thermal and electrical insulation, and to heat the sample to 1200°C at a constant rate of $1^{\circ}\text{C}/\text{s}$ while the sample is under the vacuum. Moreover, the team needed to find a way to measure the temperature of the sample from 20°C to 1200°C and needed to calibrate the electron gun and the sample holder. The sample holder needed to be calibrated so that the desorbed ions travel into the mass spectrometer. Other project objectives were to shield the turbo pump from secondary ions and to measure the desorption data with a time resolution of $2\ \mu\text{s}$. Lastly, the mass spectrometer should measure up to 50 mass per charge (m/z).

Dr. Michael Lee advised the team through the project and Sandia National Laboratories (Sandia), the client, sponsored the project. Dr. Lee is an assistant professor and analytical chemist at Northern Arizona University (NAU) researching organic electronics and photovoltaic cells. This TPD-SIMS device can help Dr. Lee and his laboratory gain the ability to analyze different materials ions. However, Sandia developed interest in this project because one of their devices was working inefficiently and resulted in bad data. The cause of this problem is still unknown to Sandia. Therefore, Sandia wanted this capstone team to create a TPD-SIMS that could test samples and hopefully find the cause of their inconclusive data.

1.2 Project Description

The project provided by the Sandia National Laboratory was to design, fabricate, and install a new sample holder and ionization mechanism. The original project description provided by Dr. Lee was 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 needed to 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 was required that the sample and the ionization source were aligned properly, and proper testing of the system has been performed. Overall, the main goal of this project was 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 was last year's TOF-SIMS capstone project. This TOF-SIMS included a vacuum chamber, an ion gun, a sample holder, an ion funnel, a flight chamber, and a mass spectrometer. The setup of the original system can be seen in Figure 1.1 below.

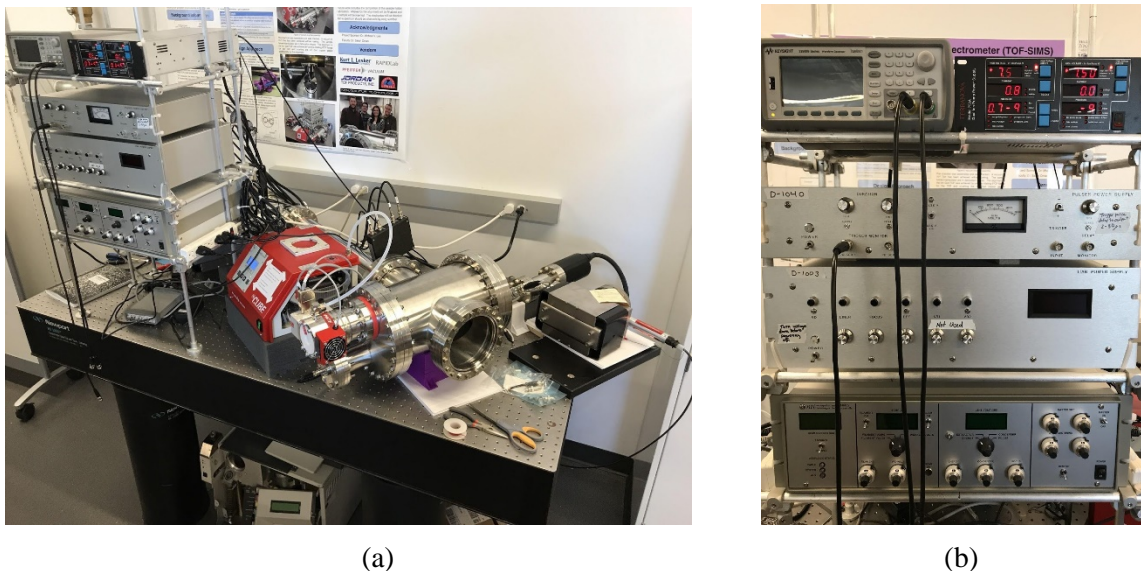


Figure 1.3: Current TOF-SIMS setup in the chemistry building. Figure (a) left depicts the vacuum chamber and pumps. Figure (b) photo shows the electronics' setup.

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 included the two ion pumps (one 30L/s and one 60L/s), the turbo pump, the stainless-steel vacuum chamber, the ion gun that discharged cesium ions at the sample, the sample holder, and the mass spectrometer. The entire system sits on top of a two-ton table that dampened any vibrations on the TOF-SIMS system. The original system was 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 performed perfectly, along with the ion pump. The original vacuum chamber held a pressure of 10^{-7} torr 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 for the cord was not broken at the end of last years' capstone. 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 did not meet any current customer requirements. It could not heat a sample at a constant rate, it could not support the new sample within the sample holder, and the ion gun could not ionize the surface of the new sample. Also, TPD was not integrated into the original TOF-SIMS in any way. However, the original system could make the chamber an ultra-high vacuum and the funnel of the original system could collect the activated ions. Overall, many modifications were 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 acted as a guide in designing and building a sample holder that allowed for temperature programmed desorption (TPD) within the ultra-high vacuum chamber.

2.1 Customer Requirements (CRs)

The team was given requirements from Dr. Lee that the design was required to meet. These requirements were provided by the client and were 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 approached the project objectives clearly and precisely and helped the team create innovating designs the client 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)

Engineering requirements are specifications that give the customer requirements quantitative and qualitative goals. The engineering requirements were created 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 must be heated up to 1200 °C.
2. The sample must be heated at a constant rate of 1 °C/s.
3. The desorption data must be measured with a time resolution of 2 nanoseconds.
4. The system must 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 must have a resistivity of at least $10^6 \Omega\text{-cm}$ to keep the electricity from reaching the chamber.
10. The total design cost must be under \$41,000.

In order to analyze the sample using TPD, the sample needs to reach a temperature of 1200 °C. This temperature allows for a TPD analysis yet will not melt the stainless-steel sample. To analyze the TPD-SIMS data accurately, the sample must be heated at a constant rate of 1°C/s. Moreover, the mass spectrometer must be able to measure a mass per charge (m/z) up to 50 m/z . This engineering requirement allows for a greater range of ions to be measured within the chamber. This range of ions will be analyzed to provide the team with a better range of data. The turbo pump must be shielded from at least 90% of the ion gas buildup within the chamber. It is crucial that the number of ions entering the pump is limited, otherwise the turbo pump will become damaged. The sample must be properly aligned within the chamber in order to maximize the number of ions analyzed. Due to the small nature of the sample holder, the tolerance of the alignment must be less than 10 nm. Furthermore, the outer chamber must not exceed temperatures of 40 °C. This will keep users safe from burning themselves on the hot metal of the chamber. Due to this safety concern, the chamber must be electrically insulated from the heated sample. The resistivity of the electrical insulation must be at least $10^6 \Omega\text{-cm}$ in order to keep the chamber safe. Also, the insulation within the chamber must have a melting temperature above 1200 °C. In the end, the entire design must not exceed the total budget of \$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

Once the system has been checked to meet all the requirements testing will begin. The tests the team need to complete will be done in the lab provided by Dr. Lee, since the device cannot be moved. The team will calibrate the thermocouples using a data acquisition software such as LabVIEW. The sample will be heated by one of the power sources control by the computer to increase the samples temperature at a constant rate. The computer will also make sure that the amount of voltage potential directly under the intake cone is zero. Keeping the potential zero will make calculations on how many molecules will be entering into the flight chamber consistent and ultimately reaching the SIMS located at the end of the flight chamber. It will be connected to a computer running a program designed to collect the data and record what molecules are coming off of the sample at a specific temperature. The computer will also be running a software called SIMION 8.1, an ion and electron optics simulator. This software allows the team to monitor the flight path of the charged particles through electric fields calculated by the software. To be able to run these programs, the computer will have a capable digital signal processing board which will be tested by the manufacturer. The electron gun, and thermocouple plus power feedthrough will also be tested by the manufacturer before arrival to the capstone team, but will be tested once installed in the system for this project.

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, but a small excerpt can be seen in Figure 2.4.1 below. The HoQ enabled a better understanding of the important customer and engineering requirements through comparison and reasoning. Each customer requirement was given a weight from one to ten with ten being the most important and one the least important. Then, each customer requirement was given a score to signify its correlation 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) was that the insulation used must have a high melting temperature. This implied 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 provided the team with two more requirements that were considered when developing designs. Overall, the HoQ provided the team with a basic knowledge of the most important requirements to meet for this project.

	Engineering Requirement								
	Project 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	Shield Turbo Pump (% efficiency) up	Outer Surface Temp of Chamber (C) Down
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

Figure 2.4.1: House of Quality Excerpt

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 development 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. This research is demonstrated in the sections below.

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. The TOF-SIMS provides elemental, chemical state, and molecular information from the surface of a solid material [8]. The average depth for a TOF-SIMS is about 1 nm making manufacturing difficult. 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 the team understand how each subsystem is connected and how each subsystem functions in the overall system. These subsystems include the sample holder, an ionization mechanism, the secondary ion mass spectrometer, data collector, and vacuum pumps and their protection. The team is tasked with designing the sample holder, possible vacuum pump protection, and the ionization mechanism. With the functional decomposition analytical methods, successful designs were 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 of 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 redesigned a sample holder, updated an electron gun, and adapted 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 need to function with the new additions, so it was important to understand each component and all of its functions within the device. The parts that were purchased include the electron gun and a turbo pump shield. However, the two pumps attached to the vacuum chamber, the computer/computer program, and the TOF SIMS were already in the team's possessions. Figure 3.3.2 shows 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.

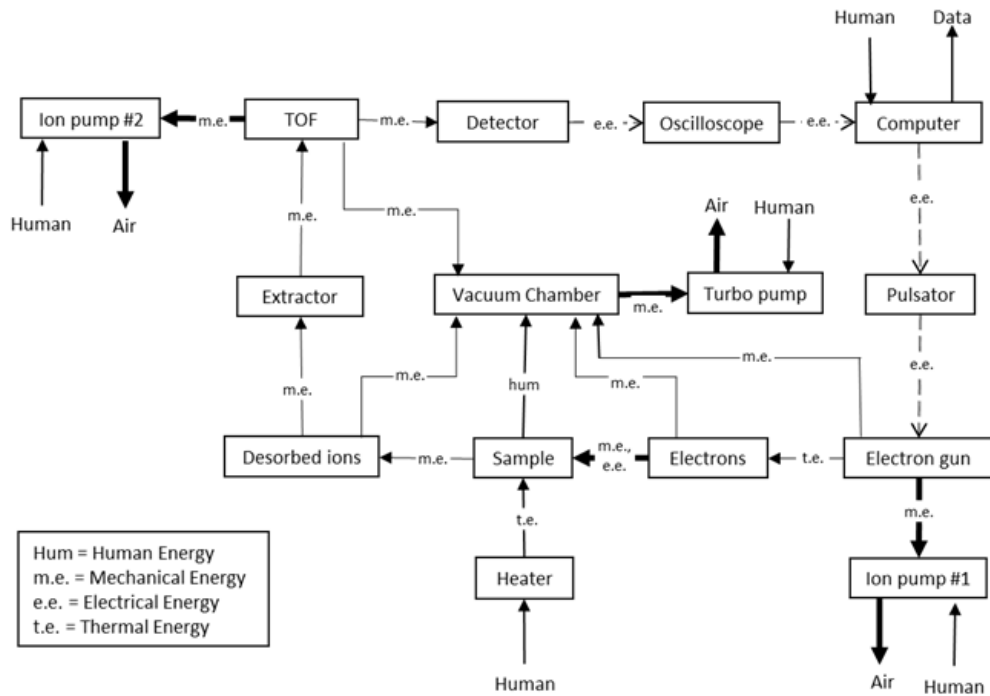


Figure 3.3.2: Functional model

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 were 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 that was being used in the TOF-SIMS was a long rectangular piece of steel. At the end of the steel plate, the actual sample holder, a small receptacle, held the material at a 45-degree angle using a spring to hold it steady and in place. This allowed the ions that discharge out of the ion gun to hit the sample and bounce into the flight chamber. This entire system pulled out linearly from the left side of the

chamber. One goal of our project was to redesign and create a sample holder that could withstand high temperatures and is electrically insulated. Because our sample is 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. Also, 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 needed 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 transluence 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. A 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 a change in sample material, these ions were too large. The team needed to research other alternatives in ionization for the new project. Alternate ionization instruments include a flood guns, a smaller ion gun, or an electron gun. This source must work at least at 70 eV, 50 μ A 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 OmniVac, 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 μ A. 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. There was no turbo pump shield apparatus in the original design. It is not known whether or not this was needed for our project. Extensive testing was done to understand its necessity. This shield is located in front of the turbo pump intake pipe at an angle that will keep out any ion gas, yet does 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 took a close look at the potential subsystems within the TPD SIMS. The team gathered to brainstorm and developed each design in order to produce innovative and creative outcomes that still met the project objectives.

4.1 Sample Holder Designs

The following sections of this report 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. 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. This design would allow the team to reuse the current sample holder while also keeping the sample stationary and insulating the arm from the chamber. However, this design might cause a high amount of stress on the sample holder arm. Also, the secondary arm holding the sample would have an electrical potential. Moreover, the electrical insulated sleeve would be hard to manufacture given the limited materials it would have to be made out of.

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

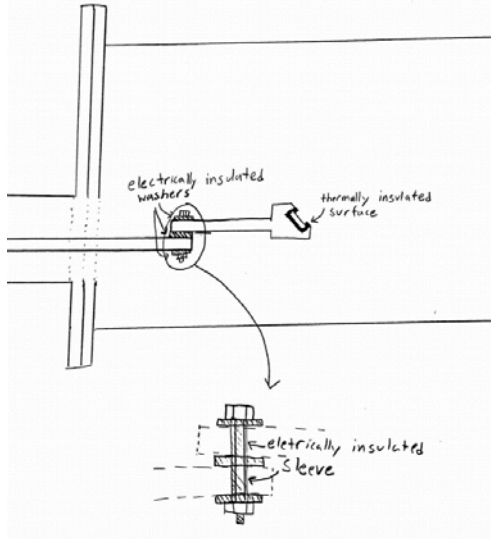


Figure 4.1.1: Insulated Sample Holder Coming in from the Side

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 collected the motor is turned until the next sample is in place and then the next test is done. The design is located in Appendix B, Figure B1. The list of pros and cons can be found in Table 4.1.2. This design would allow the team to swap samples, if required, without opening the entire chamber. However, this design would create problems with heating up each sample individually and would need more sources of power within the chamber. Another problem results from the samples being hard to align, as well as the large amount of stress put on the cantilever arm.

Table 4.1.2: Pros and Cons for Carousel Sample Holder Design

Pros	Cons
Multiple samples remain inside vacuum	Difficult to heat up each sample individually
	More weight on cantilever beam
	More power sources into the chamber
	Difficult to precisely align sample to chamber

4.1.3 Design #3: Hanging Sample Holder

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. The entrance into the SIMS is directly above the sample, making inserting the sample harder. This design makes designing the sample holder simpler because the power apparatus and the sample holder would be the same device. Due to this the team would not have to worry about electrical insulation. Some of the drawbacks of this design are drilling then sealing the chamber would be both costly and time-consuming., as well as the sample would not be able to be precisely located. 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 and time consuming
No need for electrical insulation	Not very stable within chamber, variation in sample location, not very precise

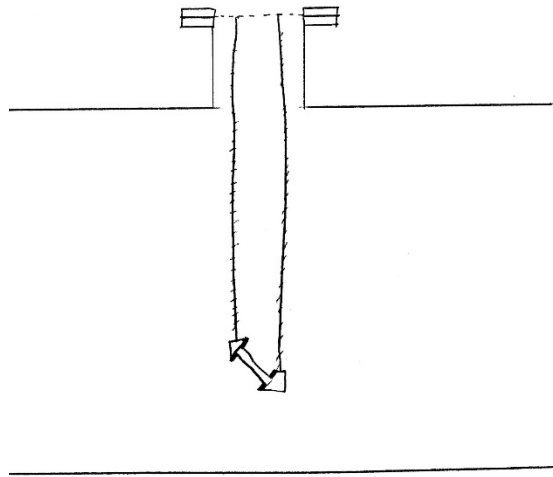


Figure 4.1.3: Hanging Sample Holder

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 laser 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. Design #4 would allow the team to use multiple samples without breaking the vacuum in the chamber and would hold the sample stationary while the experiment is running. This idea would be very expensive and hard to implement, since the sample location would be hard to get to be and the experiment would not be repeatable. Another downside of this design would be controlling the robot. It would be difficult for the team to control the robot due to the curved bottom of the chamber.

Table 4.1.4: Pros and Cons of Remote Controlled Robot with Rack of Samples

Pros	Cons
Multiple samples without breaking the vacuum	Very expensive
Holds the sample stationary while in position	The sample is subject to a lot of movement and variation in placement
	Chamber is curved, could be difficult to control robot

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]. This design would allow the team to input the samples into the main chamber without having to expose the main chamber to atmospheric pressures. This would make changing the sample easier and safer. However, this design would enlarge the length of the device causing the device to be bulky. Since Dr. Lee’s lab is small, the size of the device is important to the client. 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
Do not have to break the vacuum in the main chamber	Bulky and has space requirements
Easily and safely change sample	Expensive

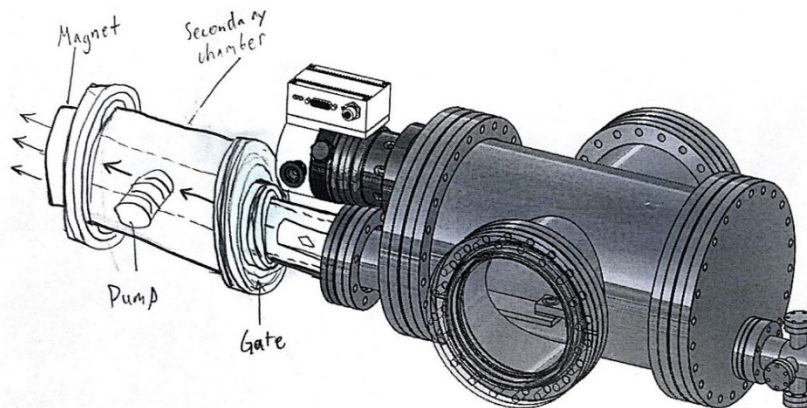


Figure 4.1.5: Sample Introduction Chamber system

4.2 Turbo Pump Shielding Designs

The following sections will describe in detail various conceptual ideas to shield the turbo pump from ion gas buildup. Three designs were conceptualized and drawn. Tabulated pros and cons were created for each design in order to analyze each design. These include only a few of the possible options available for protecting the turbo pump from any unwanted ion and any damage the ions may cause to the pump.

4.2.1 Design #6: Disconnecting the Turbo Pump

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. This design would allow the team to turn the turbo pump off during testing which would reduce power drawing from the outlet. Also, the chamber would be able to reach lower pressures while the turbo pump disconnected. However, this design would increase the workload of the ion pumps and reduce the life span of the pumps. Another negative aspect of this design is that pressure gates are expensive, and the given budget does not allow for a large expense in this area

Table 4.2.1: Pros and Cons of Using a Pressure Gate

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. This design would allow the team to keep the turbo pump on during experimentation. Also, it reduces buildup of particles or debris in the suction chamber of the vacuum. However, this design would reduce the pumping speed due to poor conductance. Moreover, if a large particle gets caught in the shield it would reduce the life cycle of the device.

Table 4.2.2: Pros and Cons of Splinter Shield

Pros	Cons
Turbo pump is constantly shielded	Reduction in pumping speeds
No accumulation of particles or debris in suction chamber	Lifecycle could be reduced if large particles get caught in shield

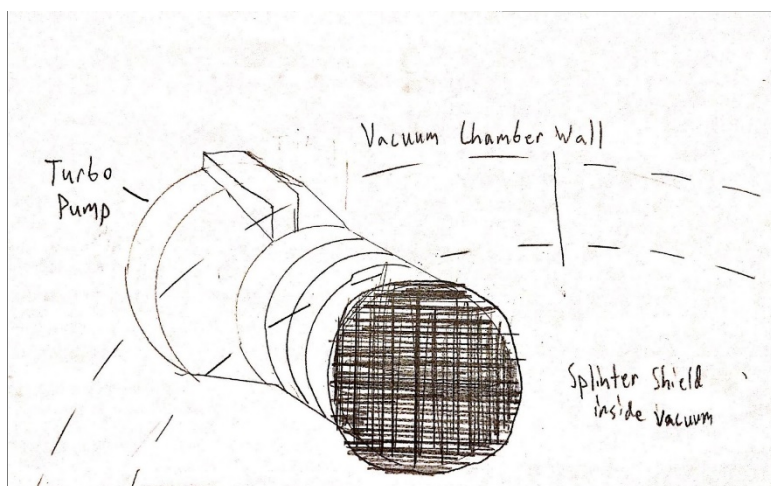


Figure 4.2.1. Turbo Pump Shielded with a Splinter Shield

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. This design would be easy to implement and fairly inexpensive. However, the turbo pump still may not be entirely protected from ion gas buildup.

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	

4.3 Electron Gun Designs

The electron gun options were limited to electron guns, flood guns, and ion guns available on the market. This limitation was due to the team not having the time nor resources to design a new electron gun, as well as a limited budget.

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

The total redesign of the system consists of all the sub designs compiled into one total design. Included in the analysis of each design are figures of the concept drawings and tabulated pros and cons for each design.

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 its 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. This design would increase the pumping speed by having two turbo pumps, as well as introduces an introduction chamber system shown in Design 5. However, this design could become off balance and fall over easily. Overall the cost of this design would increase due to having to buy new parts to rearrange the orientation of the chamber.

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.)
Increase pumping speed	

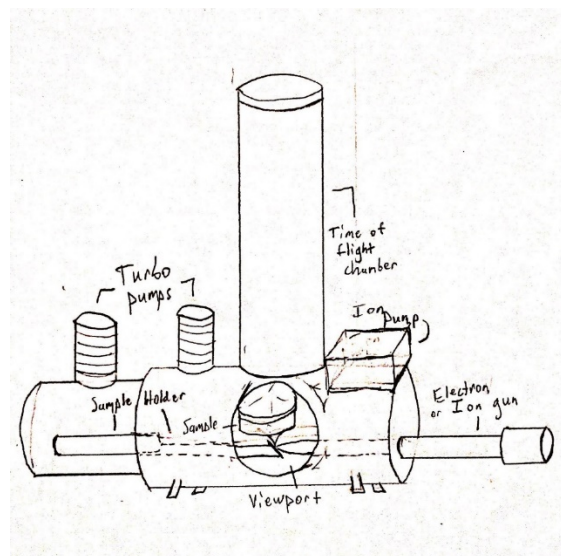


Figure 4.4.1. Vertical TOF-SIMS Experiment Setup

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 HoQ shown in Figure A1, the most important engineering requirements were thermal and electrical insulation of sample holder, as well as low cost for 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.

Dr. Lee wanted to partake in all discussions and conclusions for the design selection. It was decided with Dr. Lee once design concepts were generated, if problems were to arise, the team would use other conceptual ideas from previous meetings, and fix the problems using those designs. This means the design chosen and reported in this document may not have been the overall final design for the Spring semester of Senior Capstone. Dr. Lee was the team's client and the team wanted to make sure all designs were up to his standards and met all the engineering/customer requirements. Due to this, the team could not use any concept design tool because multiple designs were developed using prototypes constructed with common household items and several designs through CAD, as well as consulted with Dr. Lee. With the help of Dr. Lee and his experience, the hand drawn concept designs were narrowed down to determine the best designs for the project. Dr. Lee had each team member go through every purposed design and come up with pros and cons. In the end the team had discussed which designs would be created in CAD. The CAD drawings were placed in the overall system CAD to make sure it would fit in the actual system. This method helped narrow down possible design concepts and allowed the team to establish a concrete understanding of the project. The team understood this method may have not been the most reasonable way of determining the best design, however it involved our client in the way he wanted. 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 selected for purchase is 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 was 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 was approximately \$9,500 and well within budget.

5.2.2 Thermocouple with Power Wires

A thermocouple is required to measure the temperature of the sample, and power wires were 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 1,200 °C. 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.

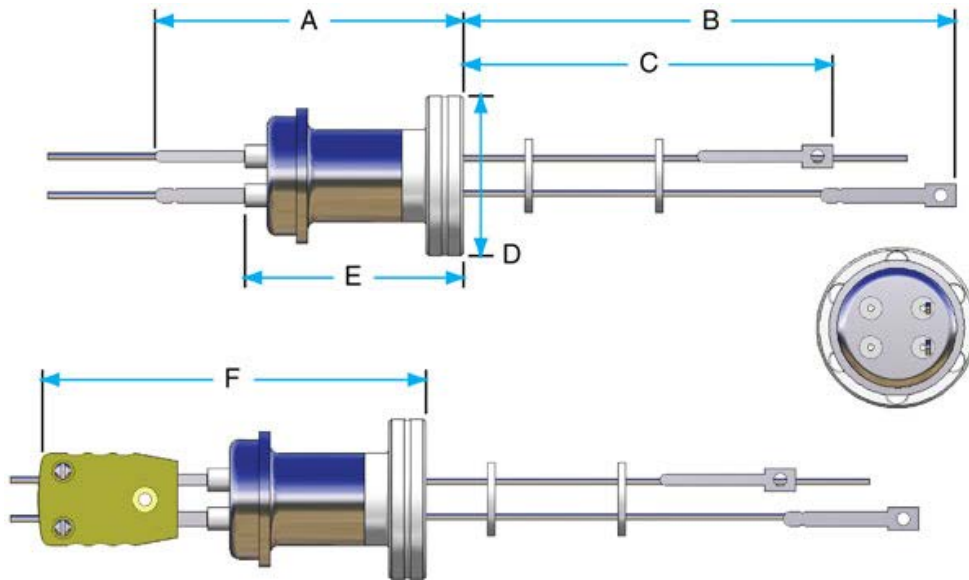


Figure 5.2.2: The Kurt J. Lesker Type K Mini Plug plus Power - CF Flanged, Single-ended thermocouple [18]

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.

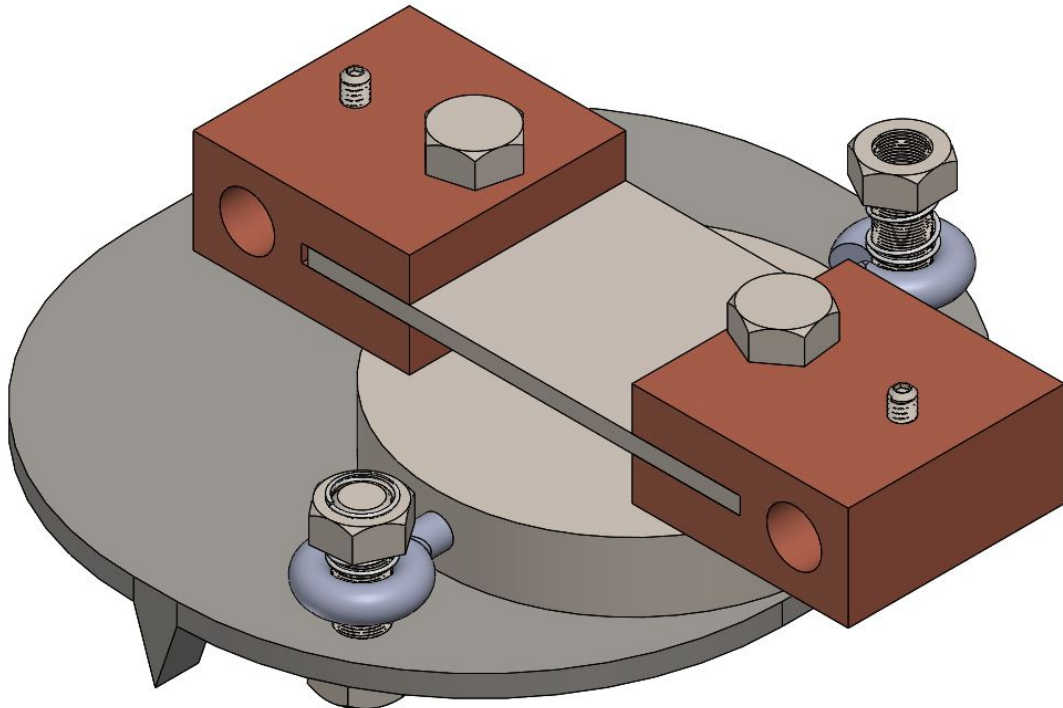


Figure 5.2.3.1: Final Design Assembly

Through the requirements provided above and the client's instructions, it became apparent 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 5.2.3.2(b). Holes were then put into the top in of the copper clamps in order to hold the wire in the clamp. Therefore, the sample needs to be modified with cutouts to allow for this screw to tighten the copper clamps. Figure 5.2.3.2(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.

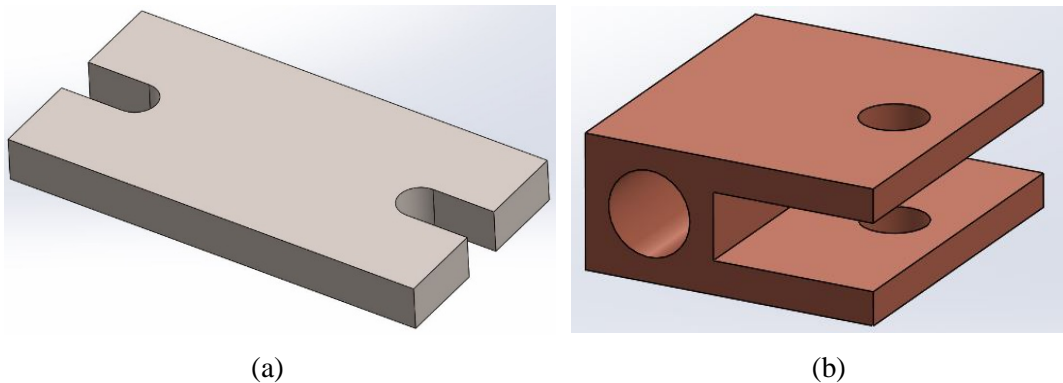


Figure 5.3.2.2: The final design for the sample and clamp. Figure (a) depicts the sample design. Figure (b) shows the clamp design.

A silicon cantilever spring will be used to hold the thermocouple to the sample. This will allow for the thermocouple to get an accurate temperature reading. In order for a material to be used in an ultra-high vacuum, it must have an outgassing rate below 10^{-9} Torr*L/sec [19]. Silicon was chosen as the material used because its outgassing rate is low enough and it can withstand temperatures up to 1300 °C without annealing. The designed cantilever spring can be seen in Figure 5.2.3.3.

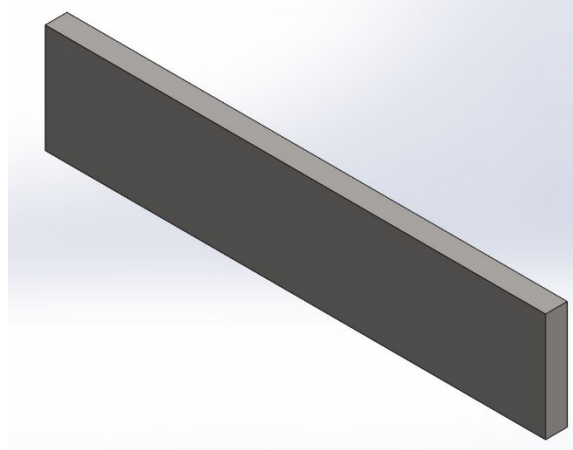


Figure 5.2.3.3: Silicon Cantilever Design

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 (Al₂O₃). Alumina was chosen based on a material analysis of various vacuum chamber allowable materials. 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 square cut in the top of the sample holder base is where the silicon cantilever will be inserted and glued down using an UHV safe adhesive. The two rounded holes on the top of the sample holder base, seen in Figure 5.2.3.4(b), are where the clamps will be bolted to the base. These screw holes are not directly above the round divots pictured in Figure 5.2.3.4(a). These indents are used for alignment of the sample holder system. The six rounded divots will cover small ball bearings located on the wedge in order to secure the alignment of the sample. The design uses six because the sample holder may need to be able to turn after manufacturing the part.

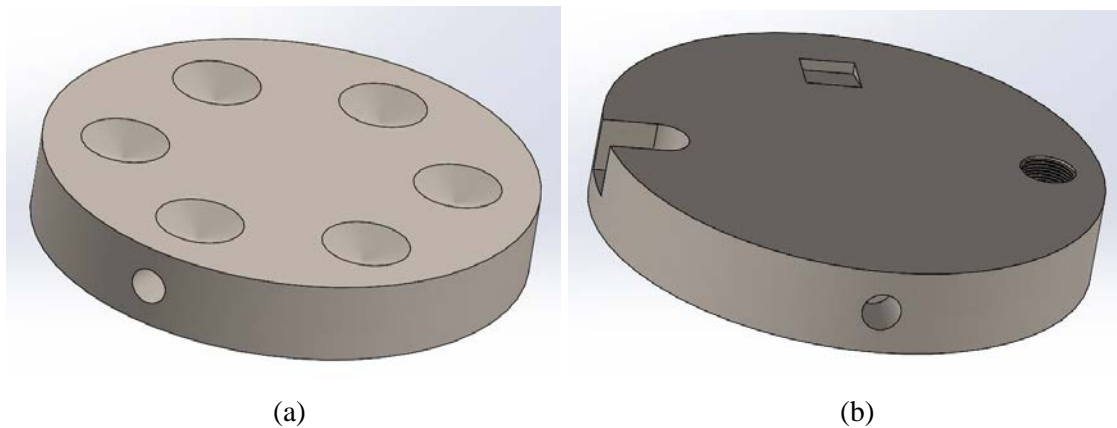


Figure 5.2.3.4: Sample holder base bottom and top view respectively. Figure (a) Bottom view, (b) Top view

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, low cost, and if machined correctly can be used in a vacuum. The middle two legs under the wedge is designed to touch the bottom of the main chamber while the two side touch the sides 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. The two holes are for the alignment screw to be placed through to hold the sample holder base to the wedge. This design can be seen in Figure 5.2.3.5.

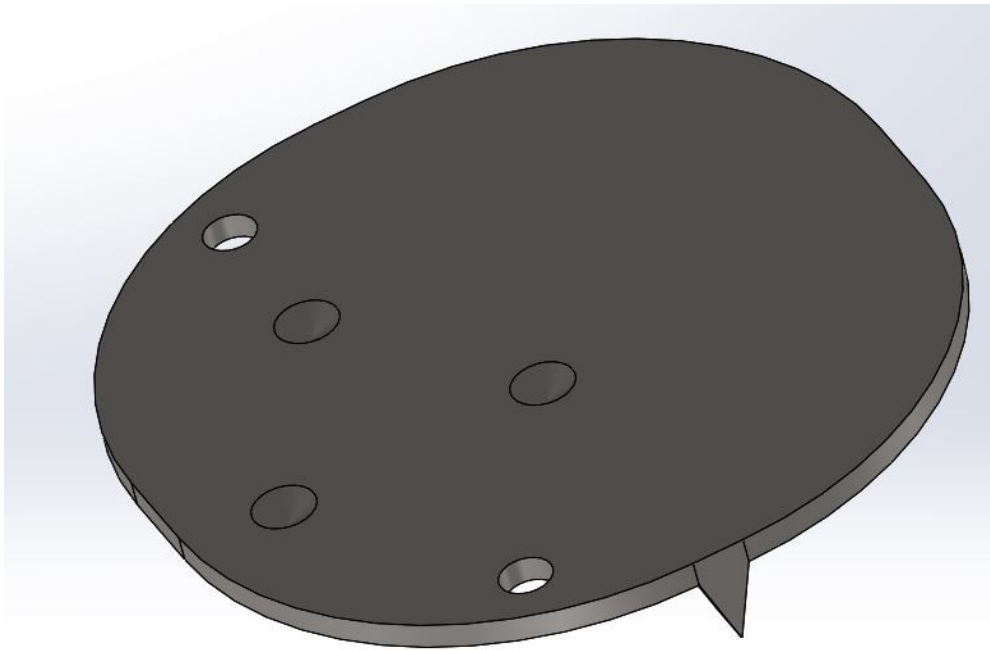


Figure 5.2.3.4: The Wedge Design

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 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, the flood gun, and the original vacuum chamber since the team will not design these devices.

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 and Figure 6.2. After the sample holder is completely finished then the team will begin experimenting with the system to test its performance.

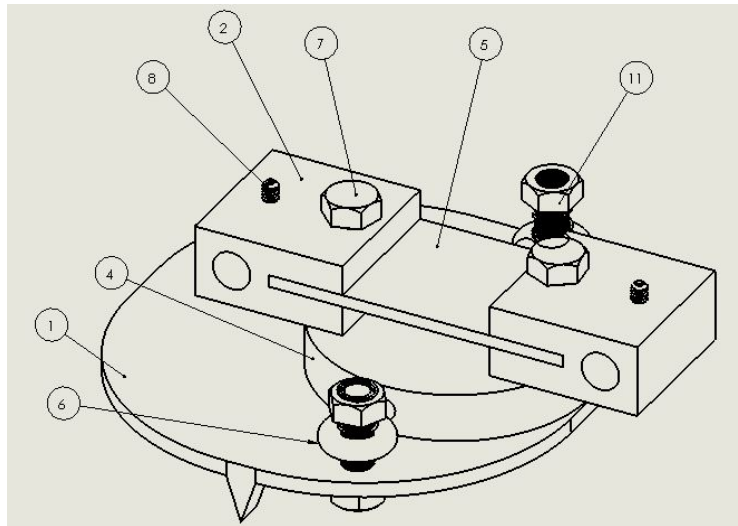


Figure 6.1: Sample Holder SolidWorks Drawing

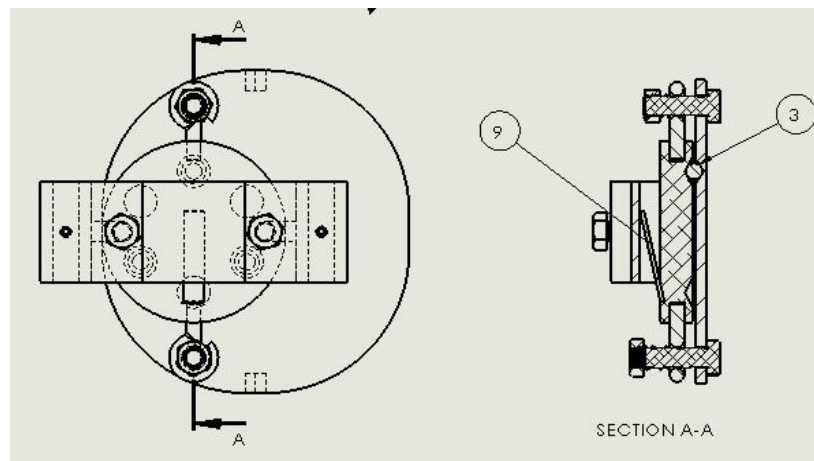


Figure 6.2: Top View with a Section Cut

Table 6.1: Proposed Sample Holder Designs Part List

Item Number	Part Name	Quantity
1	Wedge	1
2	Sample Clamp	2
3	Ball Bearing	3
4	Sample Holder Base	1
5	Sample with Cut Outs	1
6	Hook for Tightening Screw	2
7	Hex Head Screw	2
8	Clamp Power Wire Screw	2
9	Silicon Rod Cantilever	1
10	Alignment Bolt	2
11	Nut	2
12	Spring	2

A bill of materials (BoM) and a tentative budget was used for this capstone project. This was due to the nature of the parts being designed and all the different aspects of the budget. There was little information on the cost of labor for various parts of the design. For example, the Alumina insulator is a delicate part and needed special accommodations to produce the correct design. This part was 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. In the BoM an aluminum oxide rod was used as a cost estimator for the team to compare it to cost of the specialized version the team needs. 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. The BoM can be found in Appendix C Table C.1, while the tentative budget is shown 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	Kurt J. Lesker
Sample holder	\$5,000	\$4000	Various
Travel Costs	\$5,000	NA	NAU
Software	\$800	NA	SIMION 8.1
Computer w/ DSP board	\$2,000	NA	Dell
Miscellaneous	\$7,500	NA	Various
Total	\$40,850	\$14,168	

The table outlines the tentative budget the team was given by Dr. Lee. Each part or category of parts 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. Some items are not available (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 sample holder has a various for sourcing in the table above because there are many components involved and more research needs to be done to make sure the material and form of manufacturing can still be placed a ultra-high vacuum. The sample base holder will be molded and baked to create the designed component for the sample holder. One company that the team has researched is Marketech Inc. in Washington, United States. Marketech offers a wide range of capabilities when it comes to manufacturing ceramic parts. This company will be a likely choice for the base component, part 4 in Table 6.1. All nuts, bolts, and screws will be purchased from McMaster-Carr. The sample itself will be machined but the team has not chosen a machine shop as the sample has to be machined in a certain environment. This is the same for the sample wedge. The sample clamps will be machined at a local machine shop. The majority of components will be purchased, for example, the electron gun and power source, the thermocouple and power wires and power source, and the software and computer containing a DSP board. Table 6.3 below shows the dates the parts for the project need to be bought prior to, as well as roughly when they should come in. Although not all parts are listed, the most difficult ones to obtain are, the OmniVac Flood Gun FS100. This needs to be ordered relatively soon due to its 10 to 12-week lead time as shown in Table 6.3. 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 February 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.

Table 6.3: Part schedule given from manufactures

Device	Lead Time (weeks)	Buy Date	Delivery Date
Electron gun	10 - 12	December 11th	February 1st
Kurt J. Lesker Thermocouple	5	December 13th	January 10th
All other parts	2 - 3	December 15th	January 1st

7 IMPLEMENTATION – Second Semester

This section documents all design changes made before testing begins and explains any implementation issues.

7.1 Manufacturing

Once the team obtained funding from Sandia National Laboratories, the team was able to send in all the part request forms to Dr. Lee. This allowed all the necessary parts to be ordered through the chemistry department. All premade bolts, nuts, screws, ball bearings, and washers, were bought through McMaster-Carr. Also, a ceramic Macor plate was purchased from McMaster-Carr to be manufactured into the alumina insulator substitute. Kurt J. Lesker supplied the thermocouple power feedthrough, the thermocouple wire, the vacuum epoxy, the barrel connectors, and the ceramic insulator beads. Moreover, the copper power wires that will be used to connect the copper clamps to the thermocouple power feedthrough was purchased from Sequoia Brass & Copper. Sigma Aldrich supplied the coating chemicals that will be used to coat the stainless-steel wedge. However, the spray gun used to apply the coatings was purchased from Amazon. Moreover, Duniway supplied the gate valve, the zero-length adaptor, the elbow flange, and the copper gaskets. The custom-made alumina insulator and the sample are supplied by the sponsor, Sandia National Laboratory. Lastly, the custom copper clamps and stainless-steel wedge were manufactured by AC Manufacturing. The final design can be found in Figure 7.1.1 along with its respective Bill of Materials (BOM). In Figure 7.1.1, the highlighted items in the Bill of Materials represent the pieces that were custom designed by the team.

Item No.	Part Name	QTY
1	Wedge	1
2	Ball Bearing	3
3	Alumina Base	1
4	Copper Clamp	2
5	Sample	1
6	Eye Bolt	2
7	Hex Bolt (16mm)	2
8	Set Screw	2
9	Eye Bolt	2
10	Hex Nut	2
11	Washer	2

Figure 7.1.1: Drawing and BOM of Final Design

With a total budget of \$40,000 the team has spent \$20,582.59 on all parts bought thus far. This leaves \$19,417.41 left over in the budget. The total Bill of Materials and costs can be found in Appendix D. A brief overview of the Bill of Materials can be seen in Table 7.1.2 below. Table 7.1.2 illustrates the electron gun cost and travel costs were less than anticipated. The sample holder was also significantly lower than anticipated because most of the sample holder parts were custom made for an ultra-high vacuum. However, the power supplies were twice as much as the anticipated cost. This was due to the fact that the team did not expect to have to buy two separate power supplies. All other costs were within reasonable

difference from anticipated.

Table 7.1.2: Spring Budget

	Last Semester		This Semester
	Available	Anticipated	Spent
Electron Gun	\$12,000	\$9,500	\$8,838.52
Thermocouple	\$500	\$433	\$433.00
Power Supplies	\$4,000	\$2,000	\$4,300.00
Gate Valve and Adapter	\$4,500	\$2,235	\$1,982.00
Sample holder	\$5,000	\$4,500	\$1745.28
Travel Costs	\$5,000	\$1,000	\$626.32
Software	\$500	\$800	\$100.00
Computer	\$2,000	\$2,000	\$2,000.00
Miscellaneous	\$7,500	NA	\$557.47
Total	\$40,000	\$22,468.00	\$20,582.59

The next phase of the project is to test all the parts of the system. Even though the electron gun will not arrive until April 12th, the sample holder can still be placed into the chamber and tested in a pressure of 10^{-8} torr UHV environment. Also, the thermocouple and the power supplies must be calibrated with the computer. Once this is complete, the sample will then be heated up to 1000 °C using the substitute insulator. As seen in Table 7.1.3, once the team obtains the electron gun then full system testing can begin.

Table 7.1.3: Gantt Chart 2018

Task	Responsible Party	March 11 - March 17	March 18 - March 24	March 25 - March 31	April 1 - April 7	April 8 - April 14	April 15 - April 21	April 22 - April 26	27-Apr	April 28 - May 7
Midpoint Presentation	Kirsten	█								
Hardware Review 2	Colin	█								
Peer Evaluation 2	Kirsten		█							
Website Check 2	Dalton	█	█							
Poster Draft	Nikki		█	█	█					
Op/Assem Manual	Colin	█	█	█	█	█	█			
Final Report	Kirsten	█	█	█	█	█	█			
CAD Package	Dalton				█	█	█	█	█	█
Website Final Check	Kirsten									█
Ugrads Practice	All Members				█	█	█	█	█	
Ugrads Poster/Presentation	All Members								█	
Peer Evaluation 3	Nikki									█
Obtain Substitute Parts	All Members	█	█	█						
Run Test on Substitute	Dalton		█	█	█					
Obtain all parts	All Members	█	█	█	█	█	█			
Assemble Sample Holder	Nikki					█	█			
Run Tests/Collect data	Colin							█		

As stated above, the product that will take the longest to receive and test are the electron gun and the alumina insulator. Both of these parts should arrive April 12th. As soon as they arrive, all final testing will commence. Before April 12th, the team will use the supplement thermal insulator made of Macor to test the sample holder itself. The electron gun is a part of this project and is needed to conduct the TOF-SIMS TPD experiment but was not designed by team D5. Therefore, all aspects of the sample holder will be tested first. This will be done in order to verify the design and that it works appropriately. This means heating the sample to 1200 °C (1000 °C until alumina part arrives) at a constant ramp rate of 1 °C per second. The temperature underneath the alumina will also be checked to be at a safe temperature. Lastly, the team will verify that all components are functioning such as the power supplies and the thermocouple. These will also be calibrated.

7.2 Design Changes

The main changes in the design were based off of manufacturing problems, but the general shape and components of the design were only modified slightly from the proposed design. Figure 7.2.1 is the CAD drawing of the current design of the alumina thermal insulator.

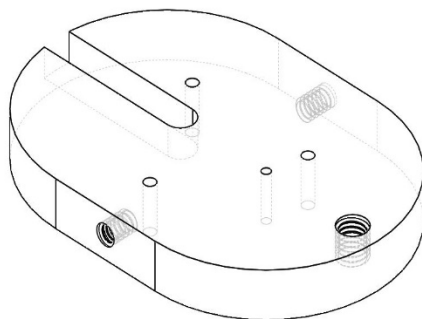


Figure 7.2.1: Alumina Insulator Updated Design

The alumina insulator was originally circular but was changed to an oval shape to prevent the bolts from blocking the electrons from hitting particles released by the sample. When the team was researching companies to manufacture the alumina insulator, it was determined that the design needed to be revised based on the small cones underneath the proposed design that were there in order to use the ball bearing alignment being a significant challenge to machine. These were then changed to through holes and reduced to only three holes as well. Another change done to the insulator was the silicon cantilever. This was found to be unnecessary and replaced with a through hole directly under the middle of the sample. Alumina, as a ceramic, is difficult to machine because the mechanical load and stress it can withstand are much lower than metals. This means that when machining, low tool head speeds and depths of cuts are necessary in order to not fracture the part. This made the price for the alumina insulator very high and have a longer lead time than expected. Do to the long lead time, the team was able to obtain a supplement part made of a similar material called Macor. Macor is a cheaper and more easily machined material with a maximum working temperature of 1000 °C instead of the 1200 °C of alumina. This substitute will be used to test the heating of the sample and verify the base will provide significant thermal insulation.

The wedge remained very similar to the proposed design. The CAD drawing of the steel wedge is pictured in Figure 7.2.2. The only difference is the cones for alignment were changed to through holes for easier manufacturing much like the alumina insulator.

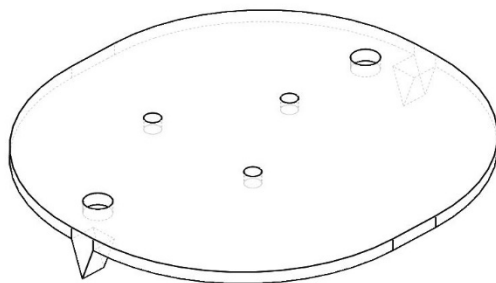


Figure 7.2.2: Stainless Steel Wedge Updated Design

The team will also add a surface coating on the top of the wedge to act as a secondary thermal insulator and prevent further heat transfer from the alumina insulator directly above it. Washers were also added to the alignment bolts to increase the area of contact with the spring and prevent any movements. In the end

the washers were used to help stabilize the sample holder. Lastly, the copper clamp design was also changed. Figure 7.2.3 below shows the copper clamp redesign.

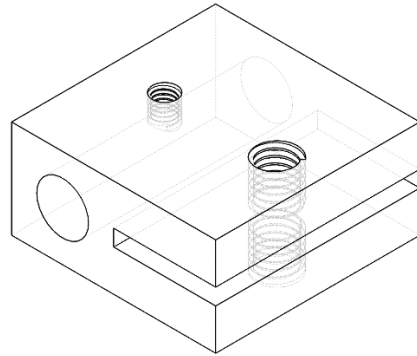


Figure 7.2.3: Copper Clamp Updated Design

Although the new design of the clamp has a similar design as the original, there were two threaded holes added to the design. The larger of the two threaded holes is used to screw down the clamp onto the sample. This will allow for better conduction between the clamp and the sample. The smaller threaded hole was designed to allow for a set screw to hold the copper wire tightly within the copper clamp.

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9 APPENDICES

9.1 Appendix A: House of Quality

Table A1: HoQ - Fall 2017

Customer Requirement	Weight	Engineering Requirement									
		Project 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	Shield 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 desorption 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	

9.2 Appendix B: Designs Considered

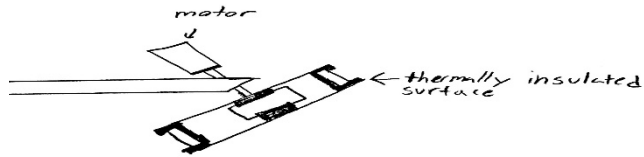


Figure B1: Carousel Sample Holder

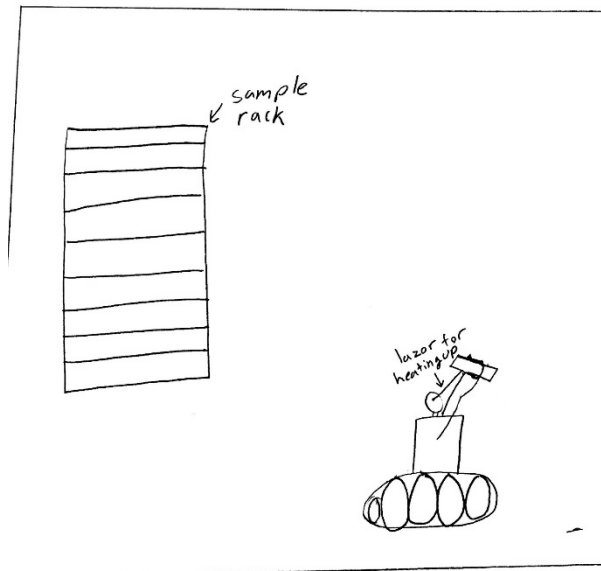


Figure B2: Remote Controlled Robot with Rack of Extra Samples

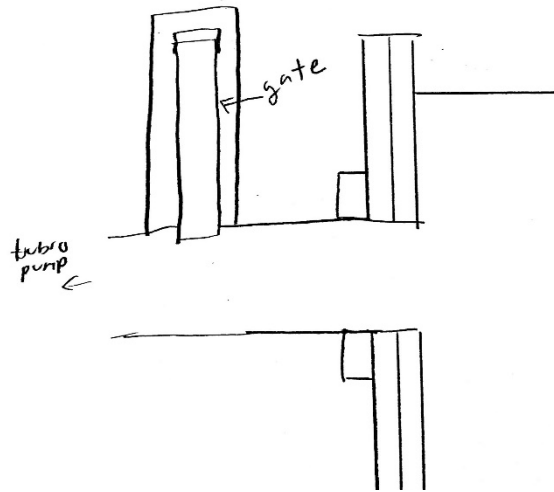


Figure B3: Disconnecting the Turbo Pump While the System is Running

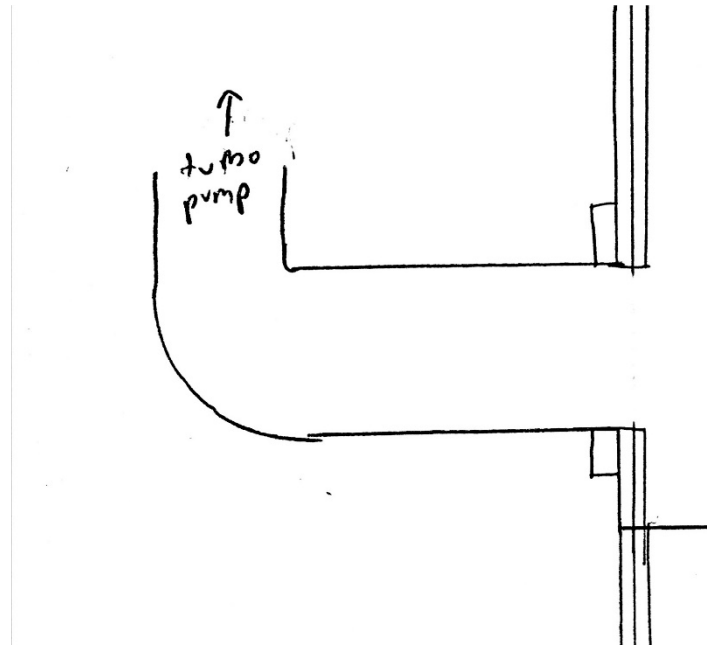


Figure B4: Curved Entry into the Turbo Pump

9.3 Appendix C: Bill of Materials-First Semester

Table C.1: First Bill of Materials

Team 04				Bill of Materials				
Part #	Part Name	Qty	Unit Cost	Description	Functions	Material	PD-SHRS D5 Dimensions	Base Cost
1	Copper Clamps	2	-	Rectangular clamps with holes drilled in in various places	Holds Sample in place	Copper	25mmx12mm	-
2	Sample	1	-	Rectangle material sent by Sandia	Used for experimentation	Stainless Steel	25mmx50mm	-
3	Sample Holder Base	1	\$416.22	Circular base with various depressions and holes	Hold clamps in position	Alumina	R-25mm	\$416.22
4	Wedge	1	-	Thin, circular plate base with two angled legs	Gives sample holder a flat surface with in circular chamber	Stainless Steel	75mm (R-35mm) x 2.5mm	-
5a	Ball Bearings Size A	3	\$0.06	Small, solid sphere	Align sample holder to wedge and allow for height variation of sample holder	Stainless Steel	D-4mm	\$5.52
5b	Ball Bearings Size B	3	\$0.07	Small, solid sphere	Align sample holder to wedge and allow for height variation of sample holder	Stainless Steel	D-4.5mm	\$7.05
5c	Ball Bearings Size C	3	\$0.08	Small, solid sphere	Align sample holder to wedge and allow for height variation of sample holder	Stainless Steel	D-5mm	\$8.38
6	Clamp/Sample Screw	2	\$0.09	Small hex head screw	Allow clamp to hold sample tightly	Stainless Steel	M4x0.7mm	\$8.52
7	Clamp/Power Wire Screw	2	\$0.29	Small set screw	Hold wires against copper clamp	Stainless Steel	M2.5 x 0.45 mm	\$7.35
8	Tightening eyebolt	2	\$1.11	A bolt with a ring shaped head	Align sample holder to the wedge	Stainless Steel	5/8"x3/4"	\$5.55
9	Spring	2	\$0.03	Small compression spring	Hold down the tightening eyebolt	Stainless Steel	D-5.3mm	\$1.63
10	Alignment Bolt	2	\$0.40	Small hex head screw	Align sample holder to the wedge	Stainless Steel	M5x0.8	\$4.00
11	Nut	2	\$0.06	Chamber assembly nuts	Tighten spring onto bolt	Stainless Steel	M3x0.5mm	\$5.55
12	Cantilever Spring	1	\$861.00	Silicon rectangle	Hold thermocouple against sample	Silicon	50 mm D-2mm	\$861.00
13	Flood Gun	1	\$10,000.00	Shoot ions	Ionizes particles on sample	-	146mm incision depth	\$10,000.00
14	Thermocouple and Power Feedthrough	1	\$258.00	Two thermocouple wires and power wires with current supply Bolt on CF flanged adaptor for vacuum chambers	Measure the temperature and heat the sample Allows for Thermocouple to attach to Chamber	-	2.75"CF 4-1/2" OD 2-3/4" 1.5 Bore 1.6" thick	\$258.00 \$109.00
15	Zero Length adaptor	1	\$109.00		thermocouple/Power feedthrough to the sample to measure temperature of sample	Stainless Steel		
16	Thermocouple Wires	2	\$3.50	K-Type thermocouple wire		Chromel+/Alumel-	0.05"	\$3.50
17	Power Copper Wires	2	\$4.73	Copper wire	Conduct current from power supply to heat sample	Copper	24-Gauge	\$4.73
18	Power Supply for Electron Gun	1	-	Power supply included with electron gun purchase	Power supply used for Electron gun to operate	-	-	-
19	Power Supply for Heating Sample	1	\$6,200.00	Box with heat capability and wire connectors	Power supply used for Thermocouple to operate	-	482mmx133mm	\$6,200.00
20	Vacuum Epoxy	1	\$52.00	UHV safe adhesive	Stabilize Sample holder to Chamber	-	7.8g BIPAX	\$52.00
21	Gate Valve	1	\$1,768.00	Vacuum compatible gate valve for turbo pump	Shield turbo pump from ion gas	Stainless Steel	ISO63-F	\$1,768.00
22	Software	1	-	UHV compatible software	Analyze TOF Data	-	-	-
23	Computer w/ DSP board	1	-	Software compatible board	Run Analytical Software	-	-	-
Total Cost Estimate:								\$19,726.00

9.4 Appendix D: Bill of Materials-Second Semester

Table D.1: Second Bill of Materials

Team DS				Bill of Materials					
Part #	Part Name	Qty	Unit Cost	Description	Functions	Material	TPD-SIMS DS	Base Cost	Link to Cost Estimate
1	Copper Clamps	2	\$295.00	Rectangular clamps with holes drilled in various pieces	Holds Sample in place	Copper	25mmx12mm	\$570.00	https://www.acrmanufact.com/
2	Sample	1	-	Rectangular material sent by Sandia	Used for experimentation	Stainless Steel	25mmx60mm	-	https://www.acrmanufact.com/
4	Wedge	1	\$835.00	Thin circular plate base with two applied legs	Close sample holder's flat surface with in circular chamber	Stainless Steel	75mm (R-35mm) x 2.5mm	\$835.00	https://www.acrmanufact.com/
5a	Ball Bearings Size A	3	\$0.06	Small, solid sphere	Align sample holder to wedge and allow for height variation	316 Stainless Steel	D-3/32in	\$8.12	https://www.mcmaster.com/96418K17L-1BJL00
5b	Ball Bearings Size B	3	\$0.07	Small, solid sphere	Align sample holder to wedge and allow for height variation	316 Stainless Steel	D-1/8in	\$8.00	https://www.mcmaster.com/96418K17L-1BJL00
5c	Ball Bearings Size C	3	\$0.08	Small, solid sphere	Align sample holder to wedge and allow for height variation	316 Stainless Steel	D-5/32in	\$9.36	https://www.mcmaster.com/96418K17L-1BJL00
6	Clamp/Sample Screw	2	\$0.09	Small hex head screw	Align sample holder to wedge and allow for height variation	316 Stainless Steel	M5x0.8mm, L=16mm	\$10.11	https://www.mcmaster.com/920290097L-1BJL149
7	Clamp/Power Wire Screw	2	\$0.29	Small set screw	Hold wires against copper clamp	316 Stainless Steel	M2.5 x 0.45 mm, L=5 mm	\$8.53	https://www.mcmaster.com/920290097L-1BJL149
8	Tightening eyebolt	2	\$1.11	A bolt with a ring shaped head	Align sample holder to the clamp	316 Stainless Steel	6-32x3/4"	\$1.37	https://www.mcmaster.com/920290097L-1BJL149
9	Spring	2	\$0.67	Small compression spring	Hold down the tightening eyebolt	Stainless Steel	D-5.3mm	\$13.65	https://www.mcmaster.com/920290097L-1BJL149
10	Alignment Bolt	2	\$0.40	Small hex head screw	Align sample holder to the wedge	316 Stainless Steel	M5x0.8, L=18mm	\$6.93	https://www.mcmaster.com/920290097L-1BJL149
11	Nut	2	\$0.06	Chamber assembly nuts	Tighten spring onto bolt	316 Stainless Steel	M5x0.8mm	\$4.12	https://www.mcmaster.com/920290097L-1BJL149
12	Washer	2	\$0.08	A small steel disk	Used so that the spring doesn't slip around the bolt	316 Stainless Steel	#10, 0.5" OD	\$8.05	https://www.mcmaster.com/920290097L-1BJL149
13	Ceramic Substrate	1	\$144.36	A small thin plate of ceramic	Acts as the Alumina base unit for the gas	Alumina	4" x 4" Sheet, 5/16"	\$144.36	https://www.mcmaster.com/920290097L-1BJL149
14	Flood Gun	1	\$9,000.00	Shoot ions	Ionizes particles on sample	-	16mm incision depth	\$9,000.00	https://www.mcmaster.com/920290097L-1BJL149
15	Thermocouple and Power Feedthrough	1	\$443.00	Two thermocouple wires and power wires with current supply	Measure the temperature and heat the sample	-	2.75CF	\$443.00	https://www.mcmaster.com/920290097L-1BJL149
16	Hex Bolt Set	25	\$0.64	Regular Hex Head bolts	Used to connect the flange to the chamber	Stainless Steel	M8x1.38	\$16.00	https://www.mcmaster.com/920290097L-1BJL149
17	Zero Length adaptor	1	\$97.00	Bolt on CF Flanged adapter for vacuum chambers	Allows for thermocouple to attach to Chamber	Stainless Steel	4-1/2" OD 2-3/4" L.5 Bore 1.6" thick	\$97.00	https://www.mcmaster.com/920290097L-1BJL149
18	Copper Gaskets	2	\$2.00	Thin circular rings	Used to connect the flange to the chamber and keep the vacuum seal	Copper	2.75" OD	\$20.00	https://www.dunwoody.com/part/61-0225
19	Elbow Flange	1	\$275.00	Large thick steel elbow bend	Used to connect the Gate connects the thermocouple/Power feedthrough to the sample to measure temperature of sample	Stainless Steel	4.5" OD	\$275.00	https://www.dunwoody.com/part/61-0450
20	Thermocouple Wires	2	\$3.50	K-Type thermocouple wire	Used to connect the Gate connects the thermocouple/Power feedthrough to the sample to measure temperature of sample	Chromel/Alumel	0.05"	\$3.50	https://www.lasker.com/newweb/feedthroughs/wires
21	Power Copper Wires	2	\$1.76	Thin, long Copper wire	Conduct current from power supply to heat sample	Copper	24", 0.26" Diameter	\$35.29	https://www.amazon.com/Art-Site-Wire-24-Inch-Copper-10-Yards/908009V-LM60
22	Ceramic Beads	20	\$4.46	Small hollow cylinder with two screws	Keeps the thermocouple wires from touching the chamber wall connect the power wires to feed through wires	Alumina	26" ID, 5" OD ID - 0.26" OD - 0.5" L - 1"	\$99.20	https://www.lasker.com/newweb/feedthroughs/ceramic-beads
23	Be/Cu In-line Barrel Connectors	10	\$13.40	Box with heat capability and wire connectors	Power supply used for thermocouple to operate Chamber	Beryllium/Copper	482mmx133mm	\$134.00	https://www.lasker.com/newweb/feedthroughs/be-cu-connectors
24	Power Supply for Heating Sample	2	\$2,150.00	Power supply used for thermocouple to operate Chamber	Power supply used for thermocouple to operate Chamber	-	7.8m BIPAX	\$4,300.00	https://www.lasker.com/newweb/feedthroughs/be-cu-connectors
25	Vacuum Epoxy	1	\$52.00	UHV safe adhesive	Stabilize Sample holder to Chamber	-	ISO63-F	\$52.00	https://www.lasker.com/newweb/feedthroughs/be-cu-connectors
26	Gate Valve	1	\$1,590.00	Vacuum compatible gate valve for turbo pump	Keeps the thermocouple wires from touching the chamber wall connect the power wires to feed through wires	Stainless Steel	ISO63-F	\$1,590.00	https://www.lasker.com/newweb/feedthroughs/be-cu-connectors
27	Software	1	\$870.00	UHV control software	Keeps the thermocouple wires from touching the chamber wall connect the power wires to feed through wires	-	-	\$870.00	https://www.lasker.com/newweb/feedthroughs/be-cu-connectors
28	Computer w/ DSP Board	1	\$2,100.00	Software control the board	Keeps the thermocouple wires from touching the chamber wall connect the power wires to feed through wires	-	-	\$2,100.00	https://www.lasker.com/newweb/feedthroughs/be-cu-connectors
29	Acet/Vacuum	1	\$40.50	Organic Compound	Keeps the thermocouple wires from touching the chamber wall connect the power wires to feed through wires	-	-	\$40.50	https://www.lasker.com/newweb/feedthroughs/be-cu-connectors
30	Aluminum Isopropanol	1	\$169.00	Chemical Compound	Keeps the thermocouple wires from touching the chamber wall connect the power wires to feed through wires	-	-	\$169.00	https://www.sigmaaldrich.com/catalog/details/catalog/entry/S15386-1000
31	Anhydrous Ethanol	1	\$59.00	Chemical Compound	Keeps the thermocouple wires from touching the chamber wall connect the power wires to feed through wires	-	-	\$59.00	https://www.sigmaaldrich.com/catalog/details/catalog/entry/S15386-1000
32	Gravity Feed Spray Gun	1	\$21.00	Purple Gravity Feed spray gun	Applies chemical compound coating	Aluminum	-	\$21.00	https://www.sigmaaldrich.com/catalog/details/catalog/entry/S15386-1000
Total Cost Estimate:								\$20,582.59	