Apparatus for Vacuum Heating of TEM Samples

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Abstract

The purpose of this capstone project was to create a sample holder to serve several purposes. A sample holder was created that can be put into the specified vacuum chamber. Additionally, the sample holder is capable of holding samples less than three millimeters in diameter and larger than 2 millimeters in diameter. The sample holder also proved capable of heating the samples to a sustained temperature of 120°C or more. This project has been a success and has met its goals.

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Introduction:

This capstone project has resulted in the creation of a sample holder that both holds and heats a sample inside a vacuum system, in preparation for Transmission Electron Microscopy (TEM) analysis. The results of TEM analysis can be greatly skewed if there are any foreign molecules on the surface of the sample to be studied. The sample holder will allow samples to be properly cleaned prior to being analyzed.

Methods:

The first step in this process was to evaluate the needs of the overall project. The sample holder is required to fit inside of a vacuum chamber. The vacuum chamber provides a near-zero pressure environment (10e-7 mbar) for heating and cleaning the sample. Heating the sample while in a vacuum allows contaminates (typically light hydrocarbon molecules) to easily be jettisoned from the surface of the sample. Therefore, it is critical that the sample holder conforms to the vacuum chamber geometry and creates a seal to maintain the vacuum.

While not in use, the opening into the vacuum chamber is closed with a nylon plug. This plug serves as the model for the design of the sample holder. The exact dimensions necessary are taken and used to configure the sample holder. This is a meticulous process because of the required tolerances of the system.

The preliminary drawings were prepared using the measurements from the nylon plug model. There were numerous changes to these initial drawings as the design process progressed although the general configuration remained unchanged. We began with the exterior geometry and designed this to meet the requirements for ease of insertion into the vacuum chamber. We added a hole running the length of the entire sample holder. We also added a counter bore that is half an inch deep on the wider end of the sample holder. The initial full length counter bore is to allow four wires to reach the end of the sample holder. Two of these wires allow for current to flow to and heat the tip of the sample holder. The other two wires are for the thermocouple which allows us to track the measurement of the temperature of the sample.

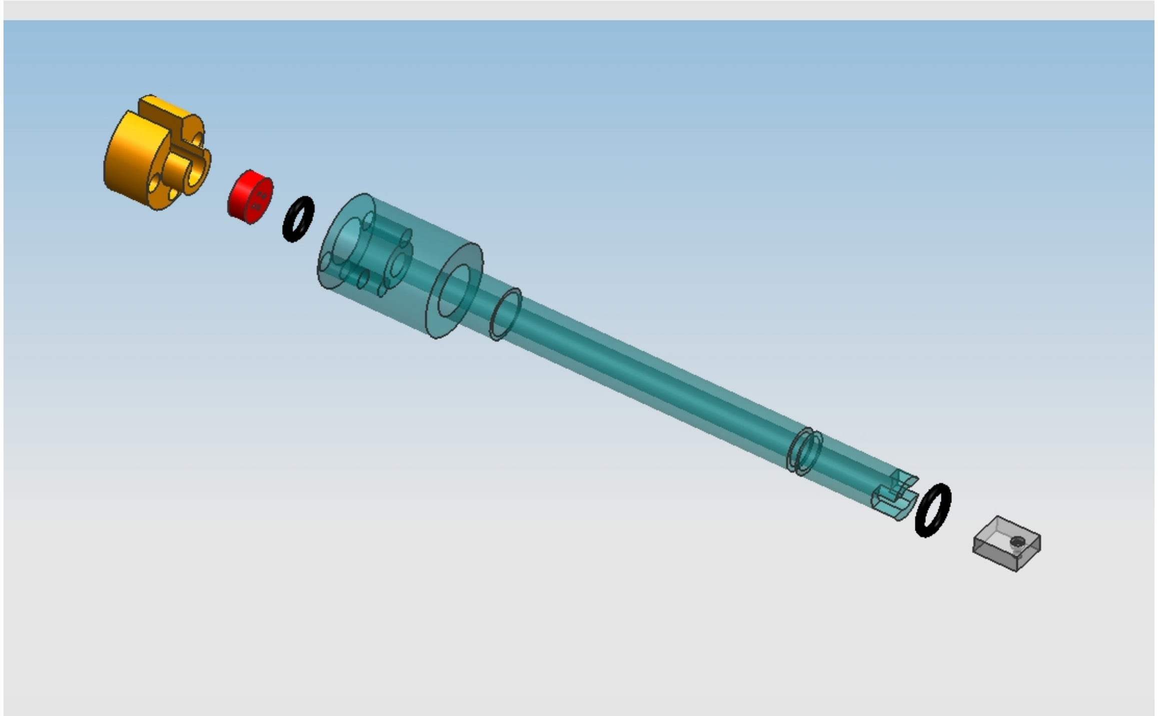

Figure 1: Exploded view of the entire assembly

The second counter bore creates a location where seals are made around the wires to prevent loss of vacuum. To achieve this we designed a disk with four small holes (seen above in red with a larger view provided below) that fits into the bore and allows the wires to pass into the first bore.

Figure 2: Disk with four holes

We also created a cap to hold the four holed disk in place. The cap has an extended part that enters the second bore and pushes on the disk. The cap also has three holes near its outer edges that allow three screws to firmly hold the cap in place.

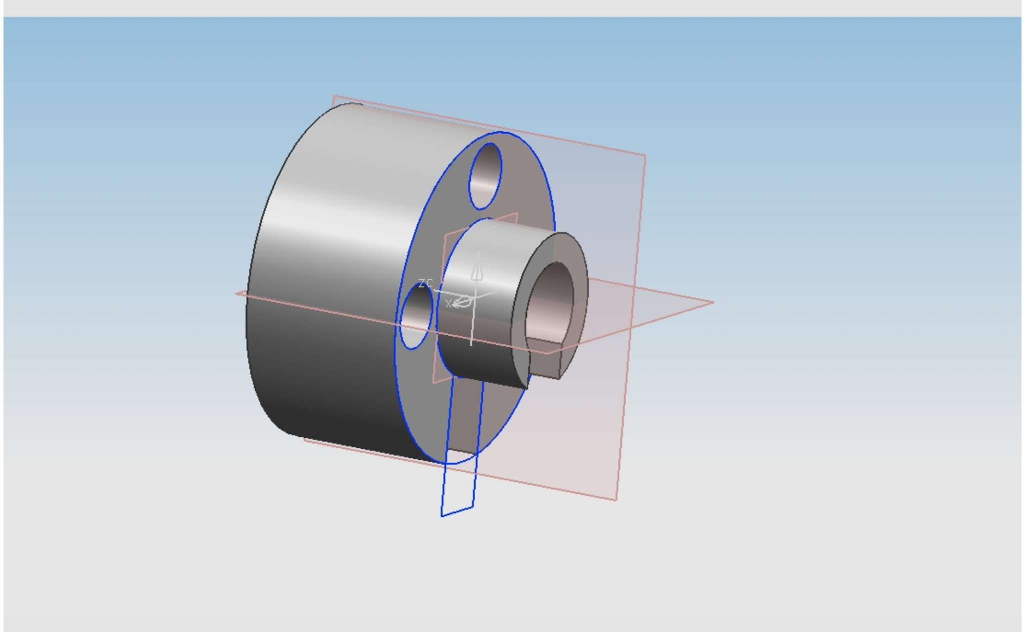

Figure 3: Cap with 3 holes for screws

The final piece of this assembly is the tip that will hold the samples. We chose a rectangular block whose width is slightly less than that of the rod part of the sample holder. In addition, the holder must be large enough to firmly hold the samples which are approximately three millimeters in diameter. Also, it is necessary that both the top and bottom of the sample are open to the environment to facilitate simultaneous cleaning of these surfaces. To accomplish this, a two millimeter hole was inserted near one end of the block with a 3.1 millimeter counter bore that cuts half way into the block. This allows the samples to rest recessed in the block with minimal room for movement.

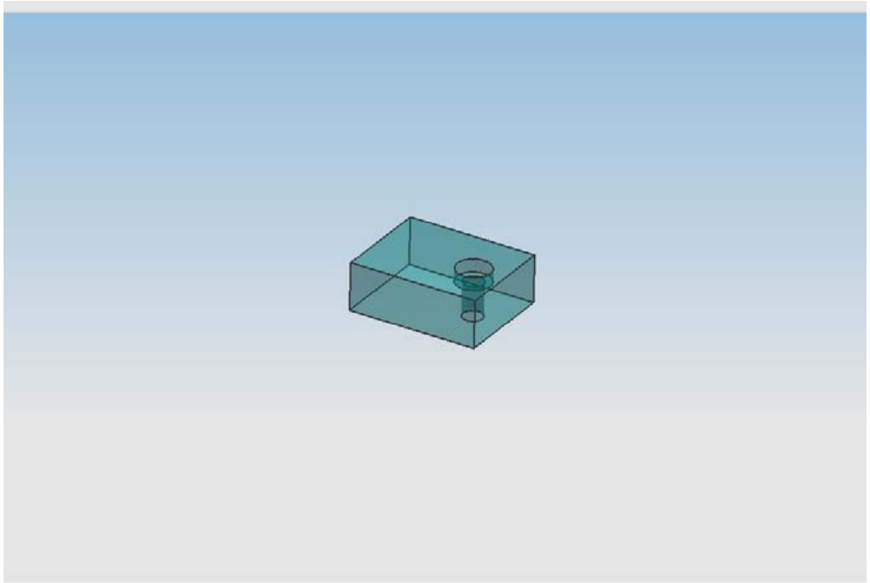

Figure 4: Aluminum block

With the initial computer designs complete, the project was submitted to the Precision Machining Laboratory to be processed. Planning for the future, three rectangular blocks and three disks with four holes were requested. This allows for trial and error in the process of optimizing the final assembly of the sample holder parts. The entire project was back from the machining lab in three weeks at a cost of $215.

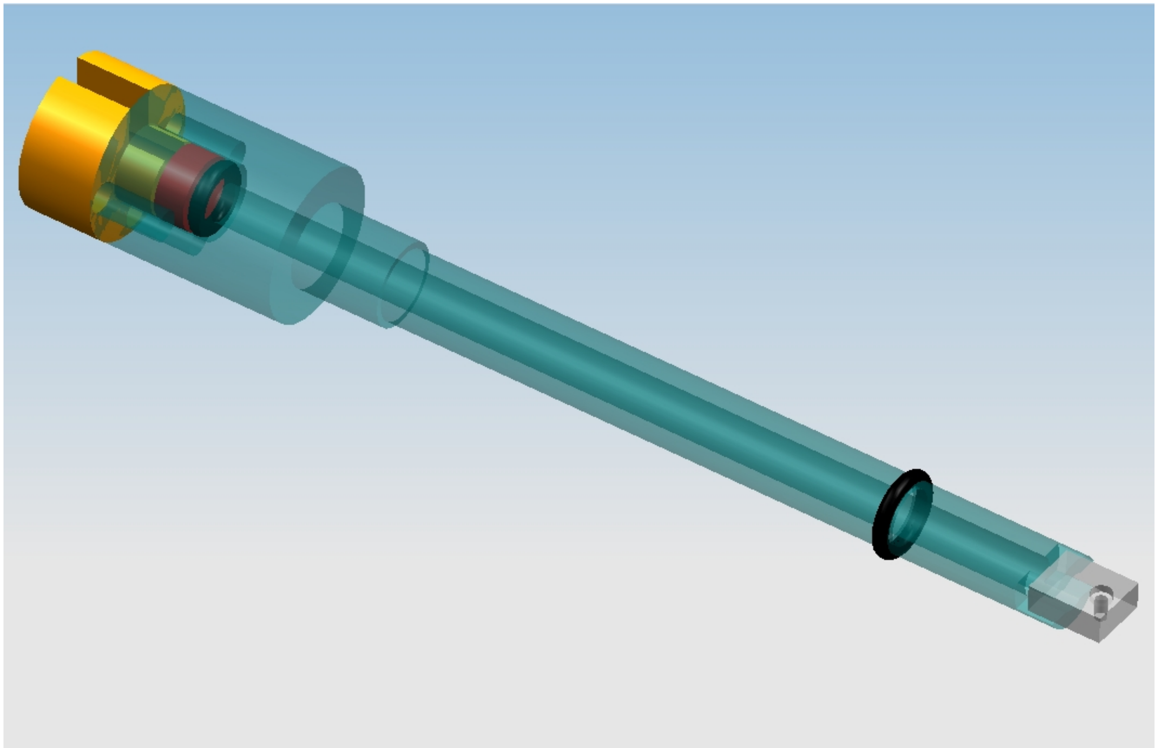

Figure 5: Collapsed view of entire assembly

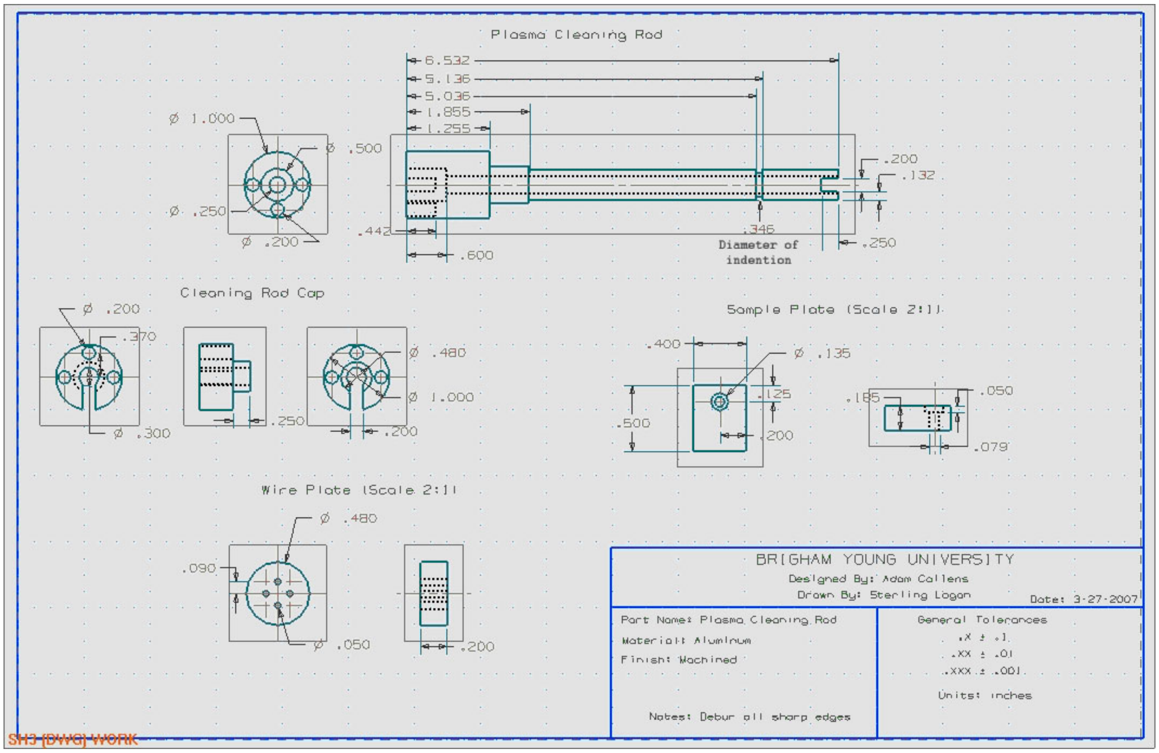

Figure 6: Engineering drawings

With all of the components available, the detailed assembly processes were addressed. A strip of zirconium is used as the heating element for the rectangular aluminum block. First, three layers of Teflon sealant is placed around the four short sides of the block. Any less than three layers will potentially melt and cause the zirconium to short across the aluminum block. Next the strip of zirconium is wrapped around the same three edges of the block. Finally the zirconium is held in place by additional two to three layers of Teflon sealant. The Teflon provides electrical insulation from the aluminum block, therefore guaranteeing electrical resistance heating of the entire strip of zirconium. A thermocouple is attached to the under side of the aluminum block to permit accurate measurement of the temperature of the block and therefore the sample.

The zirconium requires two wires for current flow, and the thermocouple also requires two wires as it measures temperature based on the change of voltage across its temperature sensitive tip. All four of these wires are passed through individual holes in the disk. An o-ring separates the disk from the location where the two bores meet. Torr Seal was applied around each of the four wires to guarantee a sufficiently tight seal.

Figure 7: Aluminum block with zirconium and Teflon sealant

The task of attaching the two wires carrying current to the zirconium strip proved to be somewhat difficult. An initial attempt of soldering the copper wires to the zirconium was unsuccessful. A suitable alternative was to insert the wires between the zirconium and the block with a press fit. Unfortunately, this increased the width of the block and therefore prevented it from entering and exiting the vacuum chamber. To resolve this we connected the copper wires with the zirconium in the space between the rod and the block and wrapped them with Teflon sealant. This setup also allows for the block to be removed and another to be easily inserted in its place if required in the future

The last step for the aluminum block assemblage was to attach the thermocouple to the face of the block. Because the entire device will be in a vacuum, it is important that the thermocouple be in direct contact with the block in order to accurately measure the block temperature. Special heat resistant tape was used to hold the thermocouple to the block. Additionally, the tape provides greater stability to the block by restricting its range of motion. With the block now attached, the cap was put in place and the three screws tightened in preparation for testing the heating and vacuum holding capabilities of the sample holder.

Mathematical Model of the Heating Process:

The heating of the rectangular block sample holder is modeled by a heat balance on the block. The heat input is accomplished by joule or resistance heating.

$$q_{in} = I^2 \cdot R$$

Where

q_{in} = heat flow into the block

I = current flow in A

R = electrical resistance in Ω

This can be written in terms of voltage drop from Ohm's Law

$$V = I \cdot R$$

$$q_{in} = \frac{V^2}{R}$$

Where V = Voltage in volts

The heat output at atmosphere pressure is the sum of natural convection and radiative heat transfer.

$$q_{out} = h \cdot A \cdot \left(T - T_a\right) + \varepsilon \cdot \sigma \cdot A \cdot \left(T^4 - T_w^4\right)$$

Where

q_{out} = heat flow out of the block in W

h = heat transfer coefficient in W/m^2K

A = surface area of the rectangular block sample holder in m^2

T = temperature of the sample holder in Kelvin

T_a = ambient air temperature in Kelvin

ε = block surface emissivity

σ = Stefan-Boltzmann constant = $5.67 * 10^{-8}$ W/m^2K^4

T_w = Wall Temperature Kelvin

The heat balance requires that

$$q_{in} = q_{out}$$

$$\frac{V^2}{R} = h \cdot A(T - T_a) + \varepsilon \cdot \sigma \cdot A(T^4 - T_w^4)$$

The heat balance at atmospheric pressure requires both the natural convection and radiative heat transfer terms on the right side. However, at the vacuum condition where the samples are tested, the natural convection term is zero since the heat transfer coefficient is zero. Dropping this term and solving first for T and later for V we arrive at the formulas:

$$T^4 = \frac{V^2}{R \cdot \varepsilon \cdot \sigma \cdot A} + T_w^4$$

$$V = \sqrt{R \cdot \varepsilon \cdot \sigma \cdot A(T^4 - T_w^4)}$$

Computations were made to determine the applied voltage, V_{app}, required for sample holder temperatures of 100 to 150°C. The resistance across the zirconium heating element was measured with an ohm meter to be approximately 30 ohms. It should be noted for future research that the resistance measured varied between 25 and 40 ohms but consistently hung around 30 ohms. This is possibly error of the ohm meter. The emissivity was assumed to be one and the wall temperature was taken to be equal to the ambient temperature of 295 K (22°C). The surface area of the holder was calculated to be 0.000365 m^2. The applied voltage was related to the calculated voltage drop across the zirconium heating element from calibration measurements at atmospheric conditions.

Calculations were made for the vacuum condition and are shown in the following table. The results are seen to be approximately linear over this range of temperatures and can be represented by the equation

$$V_{app} = 1.7 + 0.12 \cdot T$$

Where T is in degrees C and V_{app} is in volts. This equation is valid only for the temperature range of 100 to 150°C.

Applied Voltage Required for Specified Sample Holder Temperatures	
Temperature (°C)	V_{app} (Volts)
100	13.7
110	14.9
120	16.1
130	17.3
140	18.5
150	19.7

The above table is strictly theoretical, and although in range, it does not account for all possible sources of error. There are other materials in contact with the aluminum block.

The Teflon sealant is present on the block as well as the copper wires that are in direct contact with the zirconium strip. Heat is conducted along the copper wires themselves and in our formula this has been neglected.

Results:

Before testing the sample holder, a control test was conducted by setting the vacuum chamber to atmospheric pressure and timing how long it takes for it to pump down to high vacuum with the nylon plug in place. This baseline process was accomplished in two minutes. This test was repeated twice with the nylon plug replaced by the assembled sample holder. In both tests the vacuum chamber reached high vacuum at a few seconds under two minutes. This is consistent with the time required using the nylon plug. Therefore, it was demonstrated that the sample holder is able to both establish and hold the vacuum necessary for performing the required task.

Two nine volt batteries in series were attached to the zirconium strip. Heat balance calculations had shown that the approximately 18 volts that are produced will bring the sample holder near 125 °C. The target temperature for the actual sample cleaning runs is between 120 and 150 °C. The test of the sample at eighteen volts resulted in a measured temperature of 123 °C. Therefore, the test demonstrated that the applied voltage placed the sample holder within the range for optimal cleaning as predicted by the heat balance calculations.

Conclusions:

This project has been met with great success. We have been able to achieve all of the original objectives. First we are able to both create and hold a high vacuum with the plasma cleaner while the sample holder is inserted. This is crucial to being able to clean samples and prepare them for TEM analysis. Second, we are able to produce the necessary temperatures to cause contaminates to separate and be removed from the surface of samples. Both of these are crucial and therefore central to the completion of this project.

There remain opportunities for anyone wishing to make additions to this project in the future. Superior methods for attaching the end block to the rod would allow for greater stability and perhaps the opportunity to easily interchange blocks. A helpful addition would be the ability to lock a sample in place using a spring loaded clasp. Additionally, it would be beneficial to have a variable power source. One that uses current from a wall socket would remove batteries from the equation and therefore reduce the cost of operation. This would also allow for a great range of temperatures to be met in the case of unique samples with unique requirements. Along with a variable power source, the creation of a chart by experimentation comparing voltages with sustained temperatures would prove valuable.

Acknowledgements:

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