METHODOLOGY FOR OPTIMIZATION OF PARTICLE OVENS AND NEUTRAL FLUX IN ELECTRON CYCLOTRON RESONANCE MACHINES

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ABSTRACT

The Facility for Rare Isotope Beams is making developments every year to increase the beam power on the production target to support its nuclear physics program. And, at the heart of the rare isotope production, we will require continued research into the ion sources being utilized. There are currently two Electron Cyclotron Resonance machines in use at FRIB, with potential plans for a third machine as well. These machines generate a highly charged ion beam from the vapor of an isotopically enriched solid material samples. The method of production for this type of neutral flux involves various particle ovens. This study is aimed to find methods to characterize the vapor production within the scope of the ECR machines, in order to understand and optimize the capture efficiency of the neutral vapor in the ECR plasma. In doing so, a method was developed with the use of a 6 MHz quartz microbalance system to record the instantaneous flux of neutral vapor from particle ovens within a vacuum setting. A setup was developed specific to the dimensions of the vacuum chamber used, as well as the placement of the ovens relative to the plasma formation in the ECRIS. Results from this study showcase a relation between crucible design and flux across the solid angle. A Low temperature Oven (LTO) using a large stable heating mass and a simple resistive oven using Rhenium Tungsten wire were used and compared for this study, using Calcium and Zinc as source materials. The simple resistive oven, using Calcium compared deposition rates and flux change with-and without the oven nozzle. Further testing was done to compare the abilities of the LTO and resistive ovens. Zinc metal was also utilized in the oven setup to generate data on how finite temperature control influences the neutral flux recorded by the microbalance. A vapor pressure curve and theoretical calculations verified the increase observed within the system. The processes and methods exhibited in this thesis can be utilized going forward by the department to benchmark current oven outputs, as well as inspire future designs.

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CHAPTER 1

MOTIVATION

1.1 Electron Cyclotron Resonance Ion Sources

The motivation for this study stems from the beam production used in heavy ion linear accelerators. Ion sources that utilize electron cyclotron resonance have been used in a multitude of applications, with the most notable being nuclear research and experimentation. A high density of energetic electrons is generated through Electron cyclotron resonance (ECR) and confined using a high magnetic field in a high vacuum environment resulting in plasma able to ionize the particles in the system. ECR ion sources are unique in that they can generate high charge state ions from successive ionizations. The mean charge state of the ECR plasma and the resulting current that can be extracted mostly depends on the operating frequency of the ion source with higher frequency providing better performance.

Plasma is contained using two different magnet systems, and with specific configurations to create resonance frequencies. The frequency is rated in GHz, and the higher frequency correlates to better efficiency in the system and higher ionization rates. According to Geller, when the frequency of the EM wave is equal to the gyrofrequency of the electrons, the waves can transfer their energy to the electrons in a selective manner called electron cyclotron resonance (ECR) [6].

With the demand for higher beam power from the accelerator, and in particular for elements that are normally found in a solid form in nature, our focus turns towards what we can do to increase beam intensity for these elements. Current optimization pathways for most ECR machines are to increase resonance frequency inside the plasma chamber. This requires increasing the containment field and then coupling the plasma with RF power to induce resonance to ionize the neutral vapor. One of the main things to consider within the ECR machine is of course the magnets and the cryogenic cooling. These two things take up the most resources and can lead to increased frequency of resonance. Improving these systems is of the utmost importance when looking to the future of these types of machines, but it is also important to understand the rest of the processes and functions that go into making sure we have a stable quality beam.

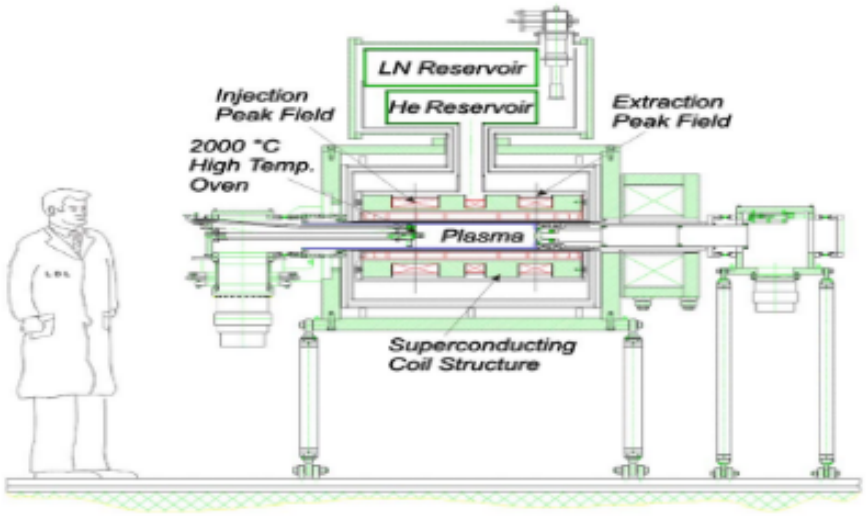

FIG. 4. (Color online) Mechanical layout of the VENUS ECR ion source. The location of the high temperature oven and the locations of the axial magnetic peak fields at injection and extraction of the ECR ion source are indicated.

Figure 1.1 Graphic of the VENUS ECR ion source. The superconducting structure is highlighted. High temperature oven is shown at the injection side, opposite the plasma and extraction side.[1]

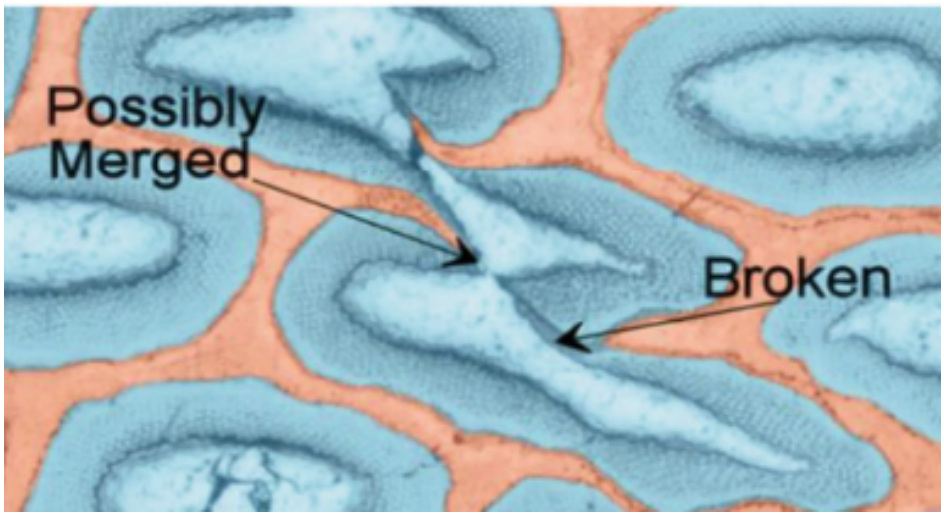

Figure 1.2 Close-up view of the Nb3Sn wire that can be used in high field accelerator magnets. Showcases the effects of the fragile nature of the wire and how it is affected by high temperature exposure. A limiting factor to higher resonance frequencies in ECRIS.[3]

As mentioned before, a main goal of this study is to work towards a way in which we can optimize the efficiency of the ECR machine. A vital source of beam creation comes from a somewhat unfamiliar place in the field of particle accelerators. The particle furnaces and crucibles that are used are an often-overlooked aspect of ion source operations. Studies done on these components can allow for less waste of precious materials and stabler beam quality. Superconducting magnet technology will continue to develop and push the limits of this ionization technology, hence why there is such a need to maintain research on these ovens. Each oven developed is, for the most part, being developed for the specific needs of the machine as well as the sample being used. Not every material you want to turn into a beam is gone about the same way, and many times there is a slight nuance to the process. The more we understand about these ovens can generate a lasting impact on the field in terms of overall quality of the Electron Cyclotron Resonance Ion Sources.

1.2 Particle Ovens

The type of oven used to inject neutral atoms in an electron cyclotron resonance system will depend on the type of beam needed to be produced. First and foremost, the vapor pressure of an element represents the equilibrium pressure of a vapor above its liquid or solid state in a closed container will determine the number of neutral particles available for ionization through a simple ideal gas law relationship. Typical vapor pressure in an ECR source can range from 1 to 10 mTorr depending on the intensity required. Moreover, for each element the vapor pressure has a unique relation to temperature that is independent of phase transition, such as melting point or vaporization point. As a result, the temperature needed to produce neutral vapor can vary widely from less than 200 ºC for selenium to 2000 ºC for Uranium Oxide. Some elements such as Calcium or Uranium Oxide will sublimate at the required temperature, while other materials such as tin would melt at a temperature significantly below the one needed for production of vapor. These requirements drive the design of the oven needed to produce neutral vapor. Once designed and fabricated, the oven is introduced inside the ion source plasma chamber and coupled to the ECR plasma as closely as possible to optimize the ionization efficiency of the neutral vapor. Heating production techniques for the ovens discussed here range from resistive to inductive, with various power transferred to the

crucible that contains the element to be vaporized.

Other studies investigated how the heating elements in the ovens work to create a temperature distribution throughout the crucible. This is important for a few reasons. First, when trying to produce an effective distribution for our material inside the crucible, we want to maintain seamless transfer of thermal energy into the crucible. And the most effective way to do this is by eliminating cold spots within our oven. Cold spots can lead to cracks in the crucible, loss of flux, and potentially even loss of material. In a paper by Haoyu Cheng, a colleague within the department, they employed a study on one of the High Temperature Ovens (HTO) utilized in the HP-ECR machine [4]. This paper lays out a thoughtful analysis of what effects the geometry of the ovens heating processes, drives neutral vapor production. For instance, in a simulation done for the HTO oven, it reveals a strong temperature disparity between the center of the crucible area and ends of the crucible. This type of research can be significant to the department's success, like contributing strategies to effectively load crucibles.

This strategy was adapted to the Low Temperature Ovens that would be used in this study as well. Using a 3D model of an LTO, mapping the temperature distribution of the crucible area and heating element would tell us a few things. Looking forward to the rest of the study requires us to have a better understanding of just what kind of temperature and thermal radiation we should be anticipating from these ovens being utilized. In an upcoming section, we will discuss the thermal radiation emitted from these types of ovens relative to the quartz microbalance system being developed. The simulation can also be used to validate the ability of the ovens. For reference, inside each of these ovens is a thermocouple that reads the temperature on the backside of the crucible area.

COMSOL Multiphysics was utilized to create the simulation seen in Figure 1.3. The heating element inside the oven runs off a power supply that when set to a certain voltage, will reach the necessary temperature inside the crucible area. This oven is modular, which means that the head of the oven can be replaced depending on what type of temperature is required for deposition needs. Essentially, there are two identical oven head pieces, one made of stainless steel and then one made

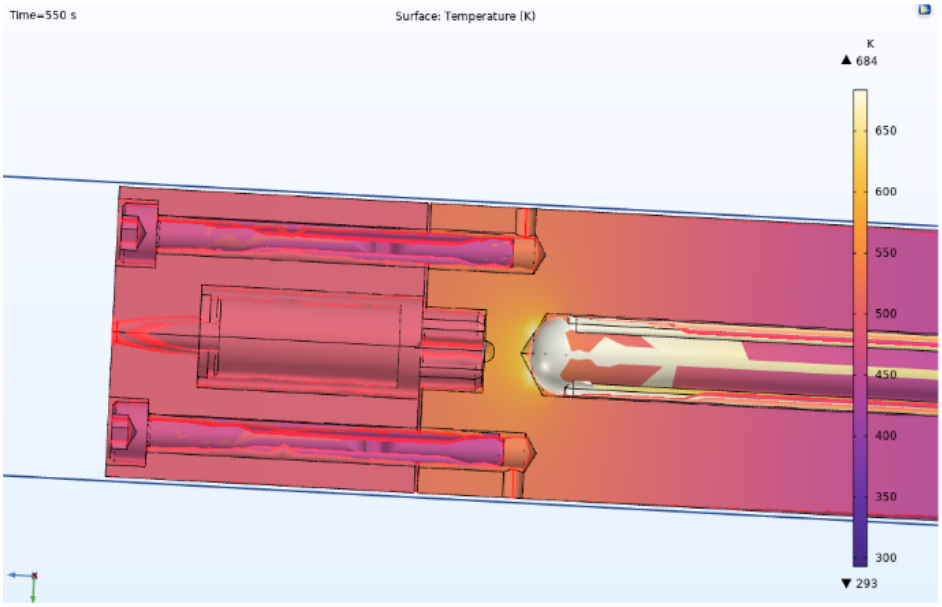

Figure 1.3 Temperature model of particle oven that is used in production at FRIB. This was generated using COMSOL Multiphysics to show the dissipation of thermal energy and temperature through the heating element to the crucible area. The head of the oven is made of copper metal.

of copper metal. The simulation was able to be done with both stainless steel and copper heads, as the software allows you to select specific sections of the oven to represent specific materials. The software also has capabilities to showcase how the temperature distribution evolves over time. This aspect of the simulation is not very applicable to the standard practices employed within the department. Since these ovens run for long periods of time on every use, they are usually brought to temperature slowly, so as to not damage the internal components. Setting the oven to 60 Volts directly could lead to deterioration of the heating element inside the oven.

There are a few things to note about these ovens being used. For instance, the length of the oven is an important factor. Although the heating element itself is small, with a typical volume around 1 cm3 length of the oven can exceed several feet depending on the ion source used. For example, for the HP-ECR the total length of the oven assembly exceeds 5 feet. Accessing the plasma chamber in a superconducting ECR requires you to go through and bypass the large cryogenic system that encompasses the chamber. All of this must be navigated through to allow a crucible, that is 1.25 inches in length, to be placed close enough as to allow for deposition into the plasma.

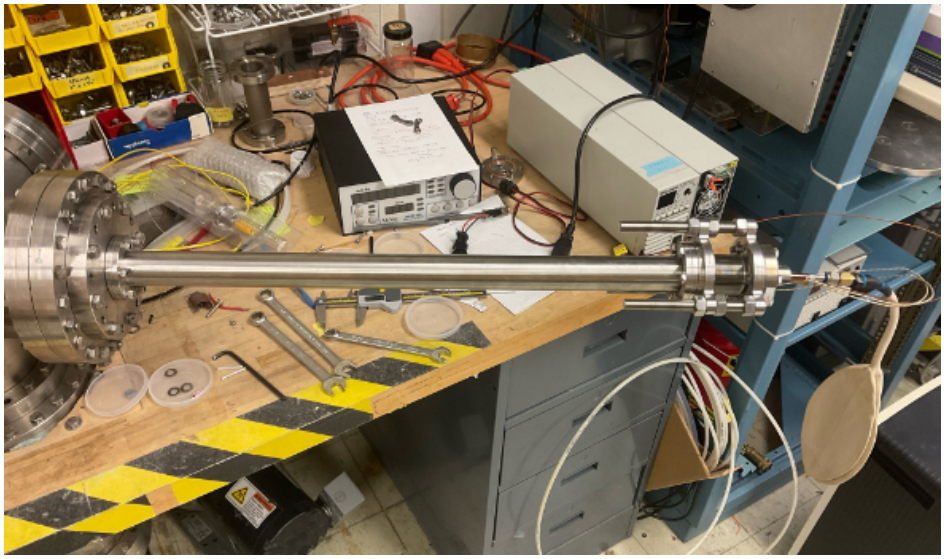

Figure 1.4 Picture of the Low Temperature Oven (LTO) inserted into the vacuum chamber. Has adjustable bellows which allows control of the horizontal depth. Balloon on the end is filled with Argon gas.

Maintaining the ovens is important, as they will be responsible for supplying the plasma with neutral material, and if there is an interruption it could cause issues with the beam production for several days. This will in turn impact the delivery to scheduled experiments throughout the lab. Issues that can impact oven operation include maintaining and regulating the oven temperature, plugging the crucible aperture, poor coupling to the plasma, quality of the material (oxidation, reaction with crucible itself). Maintaining the electrical power to the oven is usually achieved through a PID loop using the control system. However, issues can arise to maintain the temperature if degradation of the oven itself results in increasing the power coupled to the oven. For example, an electrical short or thermal short to the oven could result in degradation of oven performances or completely stop production of vapor altogether. Now, let's consider another aspect for which this study will delve much deeper into in later sections. Crucible design is a key component for these particle ovens, and much is to be said on the developments of this later. Optimizing how the vapor is produced in the crucible and is captured by the plasma directly impacts not only the performance, but also how long the experiment can last and affects the cost of buying the material used in the crucible. Typically, these materials are enriched isotopes with a low natural abundance and be

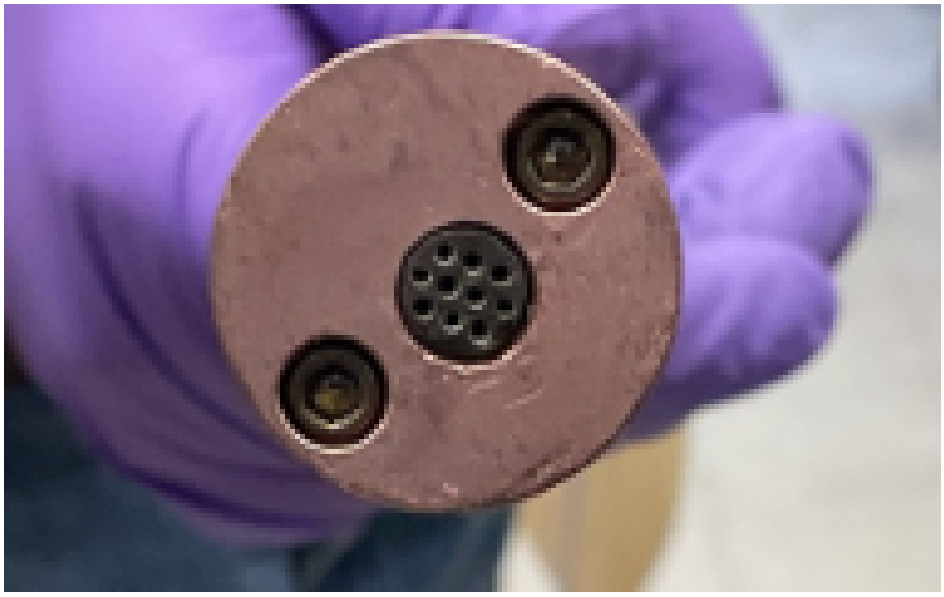

Figure 1.5 Copper head of the Low Temperature Oven (LTO). Oven exit is equipped with multiple holes. The screws on either side of the exit are used to attach the head to the oven shaft after loading the crucible.

extremely expensive (48-Ca for example) Assessing how the particle flux from the crucible spreads inside of our plasma chamber is the start of a path to better optimization for the ECR source.

1.3 Crucible Design

One thing to discuss is the difference between certain crucible designs used in this research. There can be some confusion in the later sections, so it is best to describe some of the differences now and potentially how we can improve on these designs later.

Some of the crucibles used in this study had a nozzle or a cap. This cap has a much smaller diameter for the exit hole compared to if you utilize a crucible with no nozzle. Nozzles have been experimentally used to help better collimate the vapor coming out of the oven, without changing the geometry or volume of the crucible itself. And in cases where the material will melt before reaching the desired vapor pressure, then the nozzle configuration works to prevent the liquid material from escaping the crucible and being lost within the source. The properties of the crucible are a very important consideration. Refractory materials are often used for crucible material as they tend to remain stable, even at high temperatures, and have limited chemical reactions with the material used inside. For this research, the material used for the crucible is Molybdenum. The material

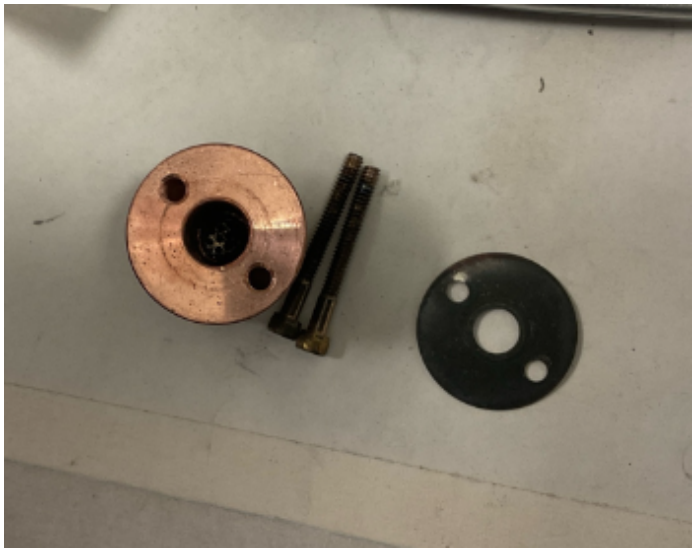

Figure 1.6 Inside of the copper head of the cartridge component of the LTO. Pictured next the copper head is the washer that makes the connection between the heating element and head of the oven, along with the screws that hold it in place.

used to make the crucible will influence the sticking time of particles inside during the vaporization process. This sticking time refers to how the particles will interact within the crucible walls, as well as the other particles of the same species. Certain species like zinc and calcium have their own thermodynamic properties which influence the sticking time. Sticking time (τ), according to the Frenkel equation [5] is dependent on the enthalpy (H) of the absorbed material.

We also make an effort within this study to recognize the effects of outside contamination during the loading and unloading process. For instance, when unloading a sample from the crucible after it has been vaporized in the oven, re-exposing it to the oxygen in the air will induce oxidation on the surface of the substance. Using a sharp edge to remove this oxidation will give us a better measurement of the sample's before and after mass.

Trying to understand more about the crucible design can be a very important aspect to improving the overall efficiency of these ovens. Uniform temperature distribution within the crucible ensures a reliable evaporation rate. While the geometry of the crucible, whether the diameter or the length over diameter aspect ratio, can help optimize the flux of neutral particles toward the plasma. This will all work to maximize their capture efficiency into the ion source. And these small improvements could result in marginal improvements, which when accumulated will better improve the sources.

CHAPTER 2

PREPARATION

2.1 Vacuum System Setup

Being able to use a vacuum system correctly is necessary for this study. Understanding how the vacuum works and using the components correctly will protect the instrumentation, as well as allow for better data acquisition. For our studies, we want to simulate a similar vacuum system that is present in the ECR ion sources. Typical vacuum arrangements include a set of roughing pumps coupled with a turbo molecular pump. The first pump ensures a transition from collective flow at high pressure to molecular flow, where the gas can be treated as an ideal gas. The turbomolecular pump contains a fast-rotating rotor that transfers the random kinetic motion of the residual gas molecules into a specific direction thus ensuring their removal from the vacuum chamber. The vacuum setup used for the studies presented here as well as ECR ion sources only require vacuum in the 10-7 Torr to 10-8 Torr and therefore does not require specialized pump as an ion pump or getter pump to reach lower vacuums.,. This involves a tandem use of a regular pump and turbo pump set up. The turbomolecular pump is an important aspect of the system due to how it must be treated and maintained. The fast-rotating rotor inside, which spins at 36 thousand Rotations per Minute (RPM), could be damaged or start to sputter if engaged at the wrong time. A point of failure in these types of pumps usually originates from the copper gasket inside the system, which if not tightened properly can lead to leaks. There is also an O-ring in the system that can form cracks if not maintained and lubricated properly. The tiny cracks that can form on this ring will decrease the efficiency of the pump. And as a result, this can prevent the system from being able to go from 10-6 Torr to 10-8 Torr, which is a large component of having accurate data collection.

The particle ovens used in production here at FRIB are manufactured to be long enough for the dimensions of the machine they are utilized in. This is for one reason, as the oven must travel far to reach the plasma chamber. The superconducting magnets used are encased around cryogenic cooling and the oven must reach past these to allow for distribution of the material into the plasma. Now, the vacuum chambers used in our ECR lab for testing are much smaller compared to the ion

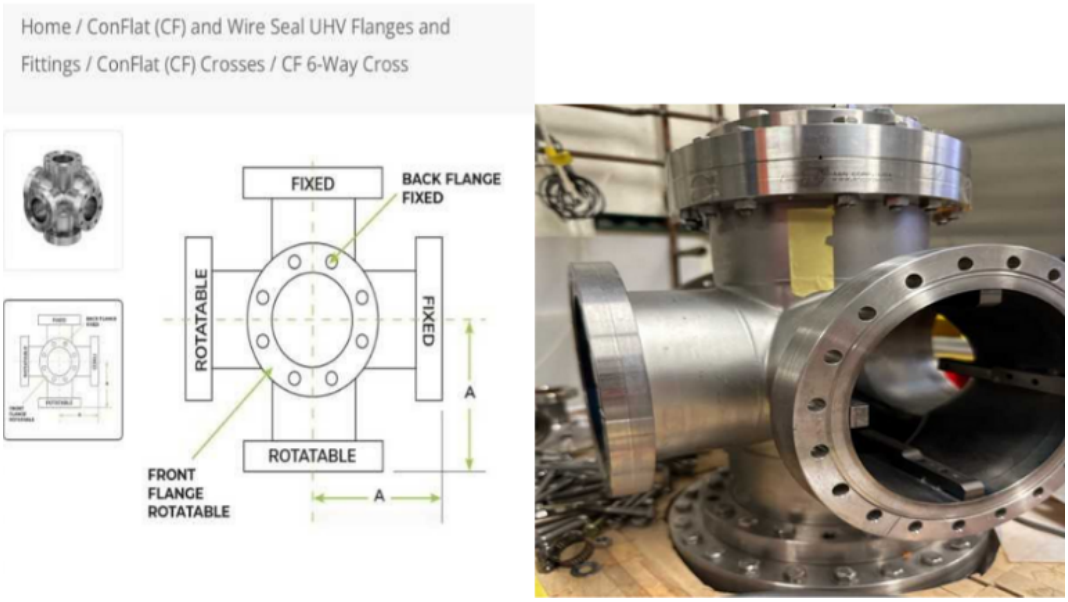

Figure 2.1 6-Way Cross UHV chamber from ANCORP. A variable from the schematic is in reference to the 6.56 inches of distance between midpoint and flange exit. Certain flange fittings are fixed or rotatable. Tube O.D. is 6 inches and the flange O.D. is 8 inches. Includes ConFlat flange capabilities.

source. This leads to having to manipulate the instrumentation used in the lab to fit around our oven setups and vacuum chamber dimensions. The vacuum chamber itself is from ANCORP, with reference number CF800-600-6X-6.56. The 6.56 at the end of the reference number refers to the distance from the center of the chamber to the flange. Furthermore, the CF800 is also a reference to the type of flange fittings it is capable of. With these being ConFlat flange fittings.

Flange fittings and feedthroughs played an important role in allowing for the instrumentation to be utilized. The vacuum chamber was fitted with five 8" ConFlat flange openings. Two of these openings would be necessary for the research study. One of the horizontal openings was utilized for the oven setup, and the necessary connectors and bellows were attached depending on the geometry of the oven. For the top opening, we would require two types of movement. Rotational and vertical adjustments would be necessary in the case of this study. A short metal bellows was added, this would allow for adjustment in alignment with the oven axis. And, the most crucial aspect of the setup was a rotatable flange, which would give the quartz microbalance system 360 degrees of freedom. The RMTG-275 flange has 2 ¾" ConFlat flange fitting capabilities, which

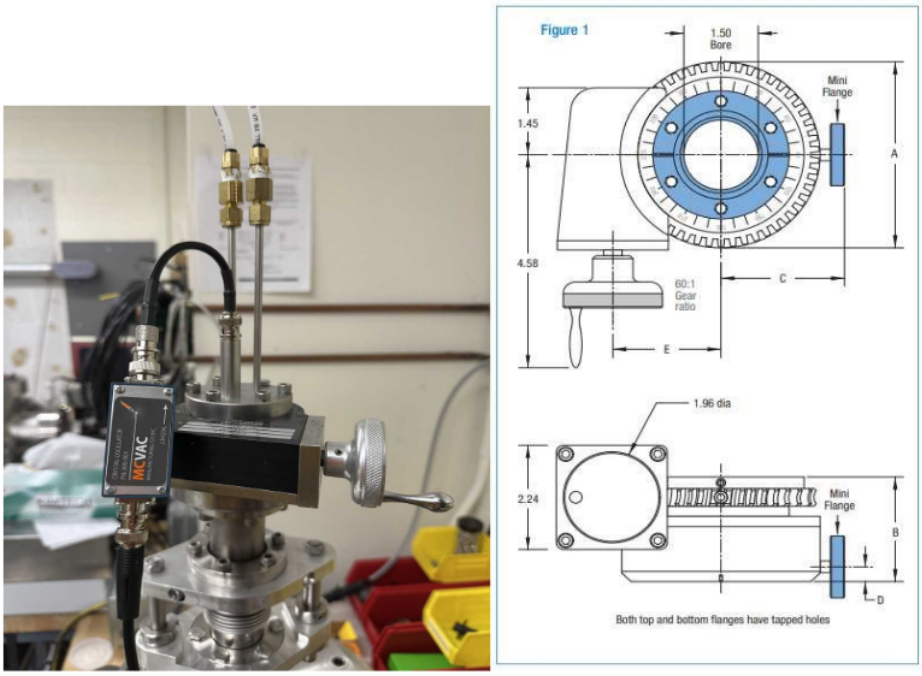

Figure 2.2 RMTG-275 flange feedthrough attached to vertical bellows. Oscillator and cooling tubes are attached to the 2 ¾" ConFlat flange from MCVAC.

would be necessary for connection to the feedthrough. The feedthrough is custom built, to allow for the cooling and BNC capabilities of our microbalance system. Rotating this flange is done by a small hand crank, which is mapped with the help of a 360º coordinate lining on the top of the feedthrough.

Inside the vacuum chamber, there is limited space to manage, especially when trying to cover a 180-degree solid angle in front of the oven. The custom cooling tubes must extend past the vertical bellows and through the 2 ¾" flange diameter of the connectors down into the chamber. After clearing this, which is almost a foot of feedthroughs and distance relative to the top of the rotatable feedthrough, we utilized a bend to accommodate the space we require to put between the crystal and the oven opening. After accomplishing this, our setup becomes quite simple. We know exactly where the crystal will sit relative to the center of the chamber. This leads us to the final step of this process. As mentioned before, FRIB utilizes different types of ovens, and these ovens have specific lengths and dimensions when being manufactured. All that is necessary for that adjustment is the necessary addition or subtraction of horizontal bellows and connectors. For example, the chamber

itself is 6.56” from the center to the 8” flange. This means that for an oven like the one used in the HP-ECR machine, which is 44.038” in length, you would need at least 37.478” of flange connections to get the oven to sit exactly in the middle of the chamber. This is exactly what was done with the help of adjustable bellows to allow for precise management of the horizontal length.

2.2 Thermal Radiation Measurements

To facilitate the advancement of the study, we must take some precautionary steps. The quartz microbalance system is fragile in terms of thermal temperature. From the manufacturer, they noted to us that the “bake out temperature” of the microbalance is around 400 degrees Celsius. This poses a problem when we want to utilize particle ovens that can reach far beyond this. But it is also important to note from a manufacturing standpoint, the temperature point provided from MCVAC corresponds to a temperature limit for the structural integrity of the microbalance system at atmospheric temperature. The crystal and cooling structure will be submerged in Ultra-High vacuum, which decreases the possibility of the total temperature reaching past 400 degrees, as noted in the thermal radiation measurement section. With the entire system in vacuum, the convection of heat is negligible, and the primary concern highlighted is thermal radiation. This radiation will have most of its impact fall on the integrity of the crystal, which is water cooled. The oven itself also has a small heating element that causes the temperature of the oven to be contained and not dissipate as easily throughout the vacuum. With this being said, progression of the study was advisable due to the cost of the microbalance and the need for protection of the technology involved.

Due to the high temperature utilized in the oven system, we reach a point where thermal energy is conveyed to the system via thermal radiation. Steffan-Boltzmann Law gives us the relation for the thermal radiation emitted from the oven.

$$\mathrm{E} = \sigma T^4 \quad (1)$$

The conductivity is relative to the emissivity of the radiation from the oven, as well as the absorption from the microbalance itself. The microbalance sensor itself has dimensions of 27 mm x 61.47 mm x 17.53 mm. There is also a gold-plated quartz crystal centering the sensor that

is responsible for the primary weighing of the sample. This quartz is the most fragile piece of the system and has a diameter of about 0.550" (13.97 mm). The size of the crystal works to our advantage regarding the thermal radiation emitted from the oven. One should note that in regard to thermal radiation, you must consider the solid angle of the radiation being emitted, same as when taking into account the particle distribution. The combination of the temperature and vacuum allows you to evaluate the radiation like a blackbody. And the crystal will absorb this radiation as a function of the angle and diameter of the crystal. The gold plating is another aspect of the crystal which makes it more resistant to the temperature change by introducing another σ into our equation from Steffan-Boltzmann. This is significant, as it allows us to be reassured in our assessment of the thermal radiation measurements.

Setting up an experiment to replicate the thermal radiation and temperature breakdown of the quartz microbalance required work with thermocouple wires and foil. As mentioned before, the quartz crystal is coated with a thin gold foil. Gold has reflective properties that are similar to the aluminum that was available, so it was used to replicate the crystal face. Using an adjustable linear feedthrough, along with a type K thermocouple, a setup was developed to place the foil as close as the actual quartz microbalance will sit. The linear feedthrough suspends the foil in a way that it is not touching the chamber walls or any conductive boundaries. A ConFlat flange fitting is normally made entirely of stainless steel. But, to electrically isolate the feedthrough, a special kind of ConFlat connector with a ceramic spacer was used. This is necessary due to how the thermocouple works and the need for it to get an accurate read on the current off the foil. The type K thermocouple is within a non-conducting sheath that runs along the linear feedthrough. The exposed end of the thermocouple then makes contact with the back of the foil, and the temperature is read out in Celsius on a multimeter.

With everything set up, an oven was inserted into the vacuum chamber that had no material loaded into it. The reason for this test was to measure the oven's own capabilities in regard to what temperature it can produce, as well as what temperature we can measure from the foil. This is a very routine check with these types of ovens. It is necessary to validate what the oven is capable

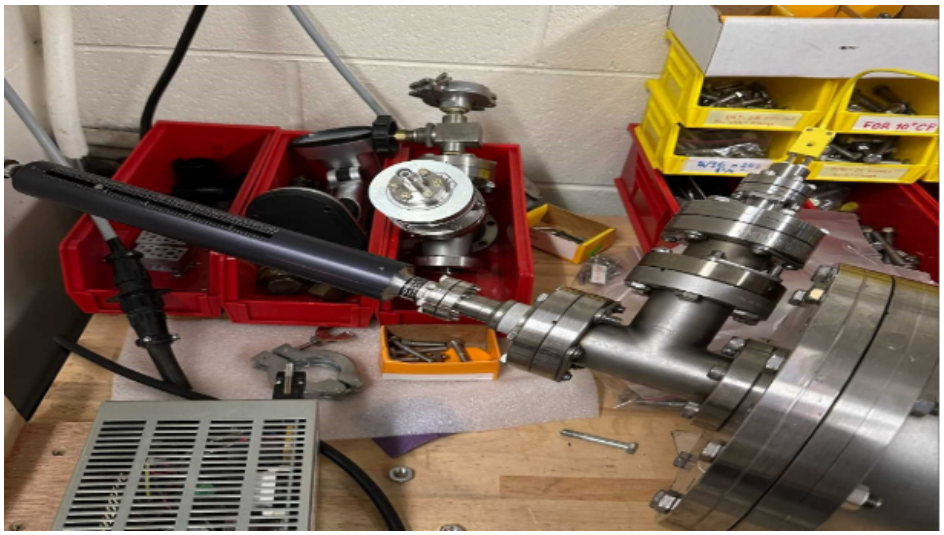

Figure 2.3 Linear feedthrough and type K thermocouple feedthrough joined with a 2 ¾" fitting. Results from this testing encouraged us to move along with the microbalance study, as the temperature recorded from this setup would be within a reasonable range for data acquisition with the microbalance.

of before inserting it into the source. The less time you have to open the source and vent, then the lower the risk of contamination on the plasma chamber walls becomes. Moisture in the air can enter the source while it is open and the oven is being replaced, and this can cause an increase in the time it takes for the chamber to be evacuated to the correct pressure. Argon gas can also be introduced into the system during the pumping process to combat the moisture in the system and speed up the process of replacing the ovens. There is a thermocouple inside the oven which reaches the back of the crucible area at the head of the oven. The oven used was of the same model used for production in the HP-ECR source, so knowing exactly how long the oven was from the dimensions made adjusting the linear feedthrough quite easy.

The results from this study included having the oven reach its necessary temperature point, and the temperature on the foil did not exceed 90 degrees Celsius. This range of temperature is acceptable and within reason for our study. Water cooling, as mentioned prior, will be used with the quartz microbalance system to maintain an effective data collection range. And, for the purpose of this study, with the type of species we plan to use for deposition on the crystal we shouldn't be worried about getting inaccurate data from the crystal.

2.3 Quartz Microbalance System

The sensor and monitor system from MCVAC [2] came with a pack of gold-plated quartz crystals, BNC connectors, oscillator, and a monitor. As mentioned in the previous section, the cooling tubes and flange feedthroughs were all custom built to meet the dimensions of the vacuum chamber we were using. The crystals themselves are replaceable and have certain lifespans. The lifespans are shown on the monitor along with the crystal status. I will delve into this some more when I talk about some complications faced that involve these crystals. But for now, it is wise to discuss how exactly this crystal can give us our flux readings.

Quartz microbalance systems work in part with a dedicated oscillator to determine the thickness accumulation on the crystal. A gold-plated crystal will resonate at a specific frequency, and when material starts to aggregate on the film, the frequency will change. This change in frequency correlates to a certain mass and thickness on the crystal. The mass is then calculated by the dedicated monitor system. The crystal and monitor systems are versatile in the fact that they can be used to measure thickness, mass, and flux. The units are defined in the manual, with the ability to interchange between them at the turn of the knob. Flux was calculated by the monitor in ng/s/cm^2 and the mass was displayed in micrograms.

All the connections need to be made correctly, and with the appropriate cables, to allow for the signal to be as accurate as possible. In total, 3 different cables were used to connect just the crystal to the monitor. The cables used outside of the vacuum chamber were normal BNC cables, but it is important to use not just any length of cable. The length of the cables is relative to the frequency of the oscillator and were provided by the manufacturer specifically for use with the monitor system.

Inside the vacuum chamber is a different type of connection. The cabling inside has a protective sheath that is temperature resistant. The connections that this cable must make are from the crystal to the feedthrough flange. The cable comes in one size and has gold metal connections on either end, to allow for accurate readings. One of the first roadblocks in this study came from an issue stemming from this exact connector. When setting up the system after all the materials had arrived, the monitor was displaying an error. The error being displayed corresponded to a “dead crystal”.

Looking at the manual, in the appendix it lists trouble shooting methods that can be deployed depending on what is seen. The crystal status and the display were blinking, which indicates a problem with the crystal. The life of the crystal, which can be visualized on the monitor via the "xtal life" button showed "dead" when pressed. This button will usually indicate a number from 0-100 and even be precise to the hundredth place. Before ruling out a dead crystal, it was indicated in the manual that a problem can arise in which the cold water from the cooling can interact with the outside air during the pumping process. This would lead to condensation and inaccurate measurements from the crystal. Water droplets will influence the resonance on the crystal face, sometimes causing an error. In response to this, the water cooling was not turned on until the vacuum had reached nominal pressure. The error still persisted, so it was time to dissect the problem piece by piece.

The monitor and oscillator system can easily be tested to validate their functionality. This is done with a test crystal, which is attached to the input side of the oscillator. The "test crystal" is not really a crystal, but a 50-ohm resistor that can connect via BNC connection. When attached, the monitor will display 0 mass and flux. It is important to note that when connected, the flashing on the monitor ceased and the rest of the functionality stayed the same. This showed us that these two components were working properly, and it was now the time to inspect the components attached to the vacuum chamber.

This type of dissection of a problem, especially with sensitive equipment like this, is done with a multimeter. The manufacturers also anticipate things like this happening, so a section of the appendix is written for this very purpose. The first step was to remove the microbalance from the vacuum chamber and assess the resistance through the feedthroughs. Resistance for the BNC and microdot connections were stable and within the acceptable measurements according to the manual. The next step involved removing the crystal holder and measuring the resistance from the gold fringes inside to the microdot connector.

Resistance measurements for this part were done delicately, to not scratch or displace the prongs. In the figure above, the in-vacuum cable is attached to the microdot feedthrough but was removed

Figure 2.4 Inside view of the microbalance sensor. Three gold prongs make contact with the back of the crystal, which is held in place by a stainless-steel crystal holder. Gold springs circling the outside of the sensor hold the crystal holder in place and allow for easy access.

for the resistance measurement. This is necessary to make sure the current flowing from the crystal to the feedthrough is not interrupted. The resistance of the feedthrough itself is measured via grounding of the multimeter on the outside of the connection site. This type of measurement will produce an overload on the multimeter, which is exactly what you should expect from a functioning feedthrough. The last component to test was the in-vacuum cable itself. This is where the problem seemed to arise, as all the other measurements aligned with the expected resistances noted in the appendix.

As mentioned before, the small microdot cable is quite fragile, especially on the ends that feed into the connectors. The microdot connection utilizes some of the same physics as in the BNC cables, hence the design is similar. A wire running through the middle of the cable is usually left with some space towards the end when connecting with the feedthrough. The ends are usually then screwed onto the feedthrough, to allow that space to be filled via the connection. The space filled in around the small wire running through the middle is what allows the cable to work like the other BNC cables. After removing the cable, it was apparent that the inside wire on the end of the cable

Figure 2.5 In-vacuum cable with gold-plated microdot connection. The inside wire tip component of the connection is crooked, causing the cable to not function properly.

that went into the flange feedthrough end, was bent. This caused there not to be an accurate signal to the oscillator. Hence, why the monitor assumed the crystal was dead and or malfunctioning.

After this discovery, I consulted with someone from a department within FRIB, who would have more knowledge on where to proceed from here. Turns out that this issue is small enough that you can bend the piece back into place and all should work just fine. This was done and the cable was put back into use. The problem was now resolved, and we could carry on with the data collection. The crystal that was utilized was in fact, not dead, and had a life percentage above 90. This was acceptable and was utilized in the testing moving forward. It is also helpful to note that most crystals installed directly from the package from the manufacturer will start at a crystal life above 90 percent, but usually not ever 100 percent. The crystal life is also a good indicator for meaningful flux and deposition from the oven being tested. As time goes on and more material is being deposited on the crystal, the lifespan of the crystal will decrease. There is also a significant temperature change that the neutral flux experiences when it moves from inside the crucible to the crystal surface. This will induce thermal energy transfer from the particles to the crystal, decreasing the life expectancy of the crystal. Hence the need for water cooling to dissipate this heat and maintain effective data collection.

For this research, we are using heavy metals like calcium and zinc. This means that the 6 MHz

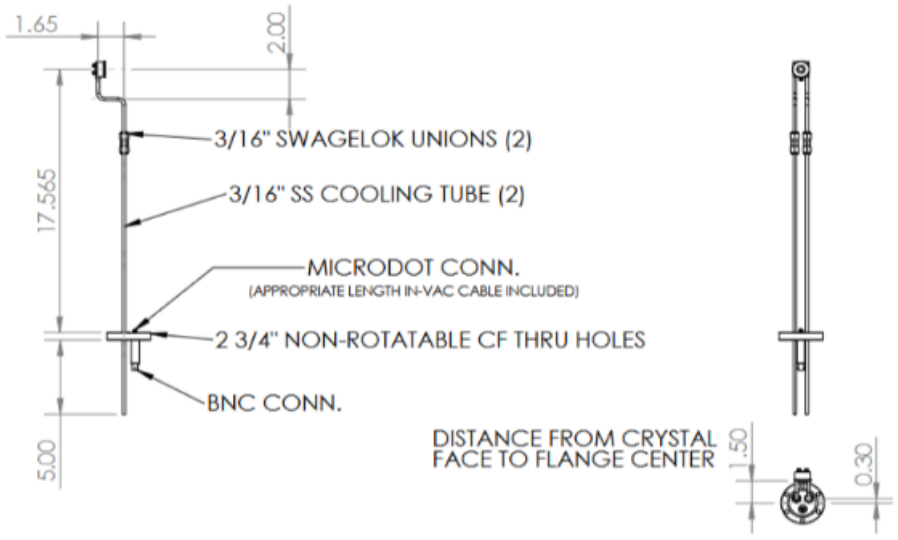

Figure 2.6 Schematic for the Quartz Microbalance Sensor System from MCVAC [2]. Indicated the length of cooling tubes utilized, along with indicators for the distance from center point of the flange to crystal face.

rated gold-plated crystals are sufficient. There are multiple types of quartz microbalance systems out there, rated to different frequencies and material deposition. For very fine material deposition, where the order of magnitude for the flux is much lower than the expected flux for this study, then a different crystal or oscillating frequency might have to be utilized. The monitor itself can be tuned to detect specific material depositions as well. This is due to the material nature of the substance being detected by the film. The monitor must be tuned to the right ratios, so as to accurately detect and calculate the flux and mass. All of this is done through the film menu on the monitor and by selecting the corresponding z-ratio and density values. All of which can be found in the appendix from the manual [2].

The final step in properly setting up the microbalance system is to ground the monitor. The oscillator and monitor work in tandem together to accurately display the measurements, so in grounding the monitor we can be sure that the current being read by the monitor is accurate relative to the change in frequency detected by the oscillator.

The quartz microbalance system is equipped with the capabilities to easily replace the gold-plated crystals. As mentioned before, different types of quartz crystals are developed for the specific

Figure 2.7 Monitor used for visualizing flux and mass readings from the quartz crystal. Important buttons include the "zero" and "xtal life", along with the configuration buttons which allow for z-ratio and density adjustments for specific materials.

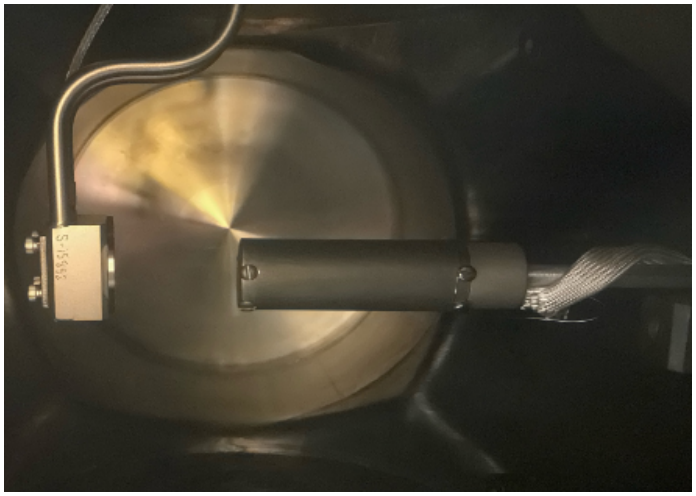

Figure 2.8 Resistive oven placed directly in front of the quartz crystal in vacuum. Distance is calculated to be about 1.5 inches.

needs of the research. Replacing the crystal was required at different periods of time in this study, and each time was due to a different reason. The first time the crystal was replaced had to be done in response to one of the issues noted in the previous sections. The root cause of this issue was with the microdot connector seen in Figure 2.5. Two different species were used in this research, and when the substrate was changed, the crystal had to be swapped as well. This was done to assure accuracy and no cross contamination.

CHAPTER 3

EXPERIMENTATION

3.1 Microbalance Data Acquisition

In a vacuum chamber, we have suspended a 6 MHz gold plated quartz crystal attached to an oscillator. This sensor works in part like a scale and a thickness monitor for particle deposition. This crystal, and all the other sensitive equipment required, was sourced from MCVAC manufacturing [2]. Once all the items required for the study were received, they were installed in the vacuum chamber with the appropriate connections and feedthroughs added. This was an important step in verifying that the system could withstand the outside pressure and maintain the low pressure within the chamber.

Water cooling is necessary for the stabilization of the reading, especially considering the proximity to the oven in vacuum. According to our calculations from the previous testing, we were certain that it was unlikely we would have any issues with readings from the crystal. Our first test with the quartz microbalance included a low temperature oven filled with calcium. The oven used was the same model used in the HP-ECR ion source at the Facility for Rare Isotope Beams. The oven was loaded with a crucible filled with 258 mg of calcium. The total weight of the crucible was 4.216 g, and the empty crucible was 3.966 g. For the first run of the testing, we placed the oven 1.5 inches from our quartz microbalance. This was calculated according to all the measurements from our vacuum setup that also considers the bend in the cooling tubes that feed into the actual crystal compartment. The oven length is also known due to manufacturer notes and diagrams. The test included varying the solid angle with respect to the oven and crucible exit. The oven works on a power supply, and the operating temperature is said to be at around 55-60 Volts. Argon gas was also introduced into the oven itself to nullify any oxidation inside the heating element. The oven was slowly brought up to temperature and when it reached 55 V, measurements were taken with the microbalance. The monitor that connects to the oscillator displays the flux and mass for the selected film and crystal. Settings were also adjusted for the z-ratio and density to account for the calcium deposition. Inside the oven, there is a thermocouple that reaches the back of the crucible

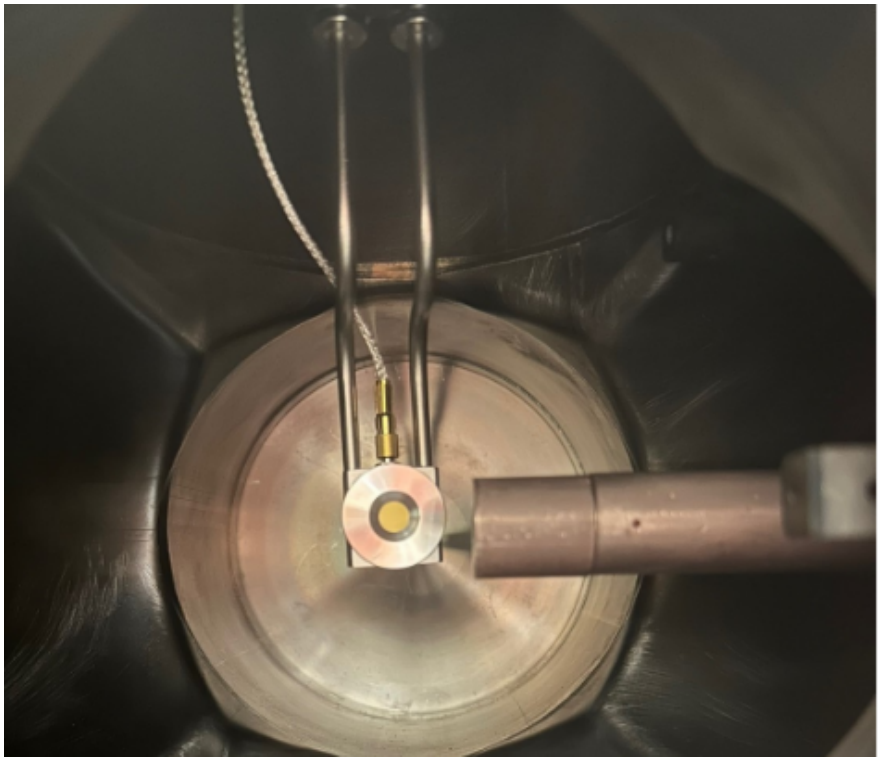

Figure 3.1 Quartz Microbalance viewed inside the vacuum chamber relative to the position of the LTO head. Gold plated crystal sits inside the stainless-steel crystal holder, with cooling tubes connected from the top, which lead to the flange feedthrough. The crystal is 180 degrees relative to the solid angle of the oven.

and gives a fair estimate of the temperature you should expect inside. This thermocouple never reached past 430 degrees Celsius, even when pushed to 60 V. This is not what we expected, and the data gathered also indicated that we may not be at the correct operating temperature. The flux from the oven was not very consistent and a lot lower than we had expected. Certain trends from watching the scale indicated that there may be an issue with calcium atoms exiting the oven. At certain angles, we would see a faster flux than normal, but once you shifted another 10 degrees the flux would almost disappear, and you would then be left with stagnant readings on the monitor. But, as mentioned before, if we were not at a high enough temperature inside the crucible then that would partially explain why the deposition was so inconsistent.

This test taught us a few different things that would impact how we carry forward. First, it was apparent we needed a reliable source of calcium flux, as this oven had not shown the ability to produce any type of meaningful data. A different type of oven, the resistive oven, would be used

for the tests on calcium and zinc going forward, as it is a very reliable form of oven. This would make sure we get a high enough temperature to the crucible area, and in turn generate enough particle flux. Secondly, flux was still recorded in the vacuum chamber, albeit low and insignificant. Nevertheless, this information was important due to how it highlights the background/noise you will see from a sensor like this. For example, while you are heating up the crucible and the vapor pressure inside the crucible starts to rise, you will notice small flux readings on the monitor. But we know from a theoretical standpoint at what temperature we need to be at to vaporize the specific material inside. From this, we can infer that what we are witnessing on the monitor is the part of the sample that was oxidized by the outside air as the sample was being loaded. In some cases, this can even be noticeable in the beam production directly from the source as the plasma and the oven are both heating up. This type of emittance is quickly filtered out within the front-end magnet systems and dipoles, not seeing much of any acceleration. But the sentiment still remains, this is an observable of the system which compounds our understanding in this study. The oven in the source will be powered on, and very small amounts of current can be generated, but at a certain temperature range all the oxidized parts of the sample are consumed and then you start to get your current relative to the material produced.

3.2 Calcium Oven Test

After discussion, we decided to move on from this specific oven and use an oven for the calcium deposit that is known‘ to be able to reach the required temperatures. A resistive oven would be chosen to conduct this part of the research. Calcium-40 was inserted into a crucible with a nozzle cap and then placed inside the oven. This oven required different length adjustments to get it to sit exactly where it is needed. At a range of 1-2 inches in front of the crystal, the bellows were adjusted accordingly. The resistive oven works differently from the previous oven that was used. This oven is most like a light bulb, in which a wire is strung through ceramic tubes that surround the crucible. The wire has a defined resistance and the power supply, which has a defined voltage and current, combine to heat the crucible. An equation can be used to derive the temperature inside the crucible, to some certainty. This is all possible since we know the resistance of the wire when

it is cold.

Data acquisition of the calcium flux from the oven was quite successful. Different voltages were utilized, and the degree of solid angle and flux was documented. The distance from the oven and the crystal was measured to be about 1.5 inches everywhere relative to the solid angle. This was sufficient to gather data from 0-180 degrees in front of the crucible exit, and to be able to observe how the flux distributed across the range. This test was repeated twice with the same species of calcium to look at the different patterns of flux between two types of crucibles. The same crucible base was used in both experiments, except for the cap nozzle, which was not included in the second test. The flux recorded in the second test showed a significant increase from when the crucible did not include a nozzle. As highlighted before, in the crucible design section, the nozzle represents a piece of the crucible that brings down the diameter of the exit. And in doing so, one might think that the material will collimate better, and you will get a tighter distribution with more flux being apparent in the solid angles closest to the middle. But, before we go into the effects of whether the nozzle is hindering production from the oven, we should assess the validity of our scientific method and get a good idea of what kind of flux we should expect from our system.

When it comes to scientific research, numerous studies and projects are built on the idea of validating and or proving the reference material found in published papers. This is one of the core principles of the scientific method, and it is also a major component of what fueled this specific research as well. The method being developed in this study can be utilized in the future by the ECR group here at FRIB to test not only flux from calcium ovens, but also different types of crucibles, ovens, and even materials meant for deposition into the source. Testing the effectiveness of the ovens before being put into production can save time and money for the laboratory.

In a paper published by French physicist [7], we were introduced to a study in which a team of researchers were looking to map out the particle distribution of calcium from a high temperature oven used in vacuum. Calcium-48 is a heavier form of Calcium that is used in particle accelerators to produce a heavy ion beam. This specific form of calcium is used a lot at FRIB, as well as multiple other particle accelerators across the globe. It is also a form of rare earth, which can be

quite expensive when looking to purchase even a small quantity. So, for the sake of the testing done in both studies, Calcium-40 was used to simulate the effects of the vaporization and flux from the oven system. The ability to be able to understand just exactly how the Calcium-40 reacts to the vapor pressure and temperature of the oven can save a lot of money for the department and protect the interests of the scientists utilizing the beam. The more information you have on the effects of the calcium flux from the oven, the better equipped you then become to advocate for optimization on the functionality of the oven.

In the paper by Leduc, they utilized a setup similar to ours, with some key differences having to do with their particular quartz crystal. The crystal used for their microbalance was rated to 3 MHz, while the one used in this study is rated for 6 MHz. The rating of the frequency is arbitrary towards the comparison of data acquisition, as the frequency of the crystal oscillations are relative to the specific standalone oscillator included with the monitor. The monitor then interprets the output from the oscillator to determine and display the mass readings. Going further, oven distance from the crystal was also made to be like the distances used in the reference paper, as we wanted to replicate a portion of their experimental results with our own setup. Replicating the results, or showing that we can't replicate them, will go a long way in telling us about how we can further improve the system of data collection. It also might even tell us something about the oven itself. Just like how our results from the previous section highlighted the lack of calcium production from an oven that struggled to get up to temperature.

For our first attempt at data collection with the Low Temperature Oven, we struggled to see little of any flux be produced with our microbalance. With this we returned to a reliable method, the resistive oven. These ovens are much less sophisticated than the LTO's and, as mentioned before, rely on a power supply tuned with respect to the voltage and the resistance. The power is generated, and in turn the heat produced from the wire will create our temperature according to the cold resistance measured before the oven is powered on. In **Figure 3.2**, it is important to note the legend on the right is in reference to the voltage of the power supply, and when calculated will refer to two distinct temperatures.

The method for which we took our data is quite simple. After the pressure of the chamber reaches the range of 1E-8 - 1E-9 Torr, we can safely start the process of bringing our oven to temperature. The pressure, which is read from an ion gauge inside the chamber, will fluctuate some as the temperature of the oven increases. This is to be expected and can also be used as a tool to properly gauge the appropriate times to increase the power. It is important to be patient and maintain the stability of the pressure in the chamber. It can take a few hours to get the oven up to temperature, but once we have set our voltage to its desired range, we can allow the amperes to stabilize and collect our data.

At around 7 volts, we are starting to see some particle flux from our oven. It is now when we set it to 7.4V and recorded the data as seen in **Figure 3.2**. Starting at degree 0 on our rotating flange, we recorded the flux and mass numbers which appeared on our monitor. In doing so, we set the flange to the correct degrees, wait a minute or two for the system to start to stabilize and then record the value. This was done every ten degrees for this moment in particular, but going forward the value was recorded every 5 degrees. Once the values at 7.4V were recorded, we then increased the voltage to 8V and moved the microbalance out of the way. With the microbalance set back to zero degrees, another set of flux values was recorded.

It is important to note in the figures we do have some vertical error bars, which denote the range of uncertainty with our collection methods. When collecting our data, the flux calculated and presented on the monitor will change relative to the time it takes for that value to be recorded. This is one of the main reasons why it was important to take time between each degree of data collection, as noted in the section above. Once the system stabilized, a number with tenths accuracy would be displayed, and this number would almost always fluctuate by a certain value. This value, which is 0.9 ng/s/cm^2 was a number like the same number we had seen from our background values. So, from here, it was safe to assume that the number from which we deviated by 0.9 from, was most likely our actual value of flux. You can also note this, by looking towards the very ends of our figure, and you will notice that specific, almost negligible background value.

The results from this initial testing with the calcium oven were quite promising. Due to what

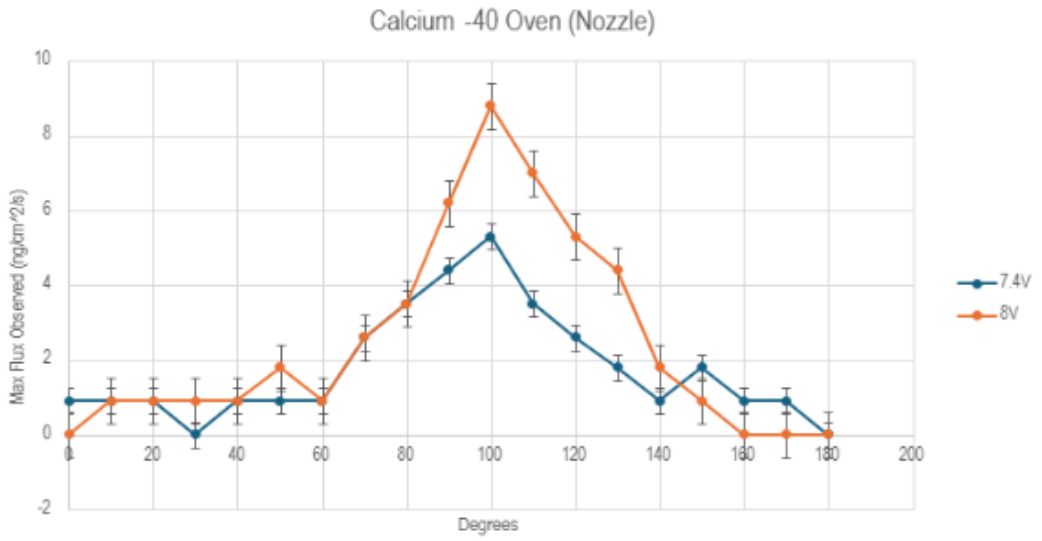

Figure 3.2 Graph of flux from a resistive oven that shows a maximum flux at around 100 degrees of solid angle. Data was recorded every 10 degrees of separation. Note the title of the graph is in reference to the addition of a nozzle on the exit of the crucible.

we were able to gain from our data collection, we wanted to alter a specific variable of the crucible design to see if it would induce more flux from our oven. The nozzle on the end of the crucible would be removed and the testing would be done again. This next set of testing will be able to tell us a few things. Our microbalance readings will be able to tell us from a numerical perspective, just how much our flux has changed. It will also indicate to us, whether it influences the collimation or spread of the neutral flux as it exits the crucible.

The crucible with the nozzle was removed from the resistive oven and the weight of the calcium-40 inside was measured. When removing the calcium sample from the crucible, you will quickly notice that the color of the sample is much different from when the sample was inserted. This is due to the oxidation of the sample that occurs, which is why certain methods are in place to limit the oxidation of the samples. The oxidized pieces of the sample can be scraped off and then a final measurement of the calcium-40 sample was recorded. The crystal in the microbalance system was removed and the deposited calcium that had formed on the crystal was removed with an alcohol solution. The crystal still had more than enough life expectancy to be utilized again. For the next set of data collection, we omitted the nozzle and utilized the same molybdenum crucible body.

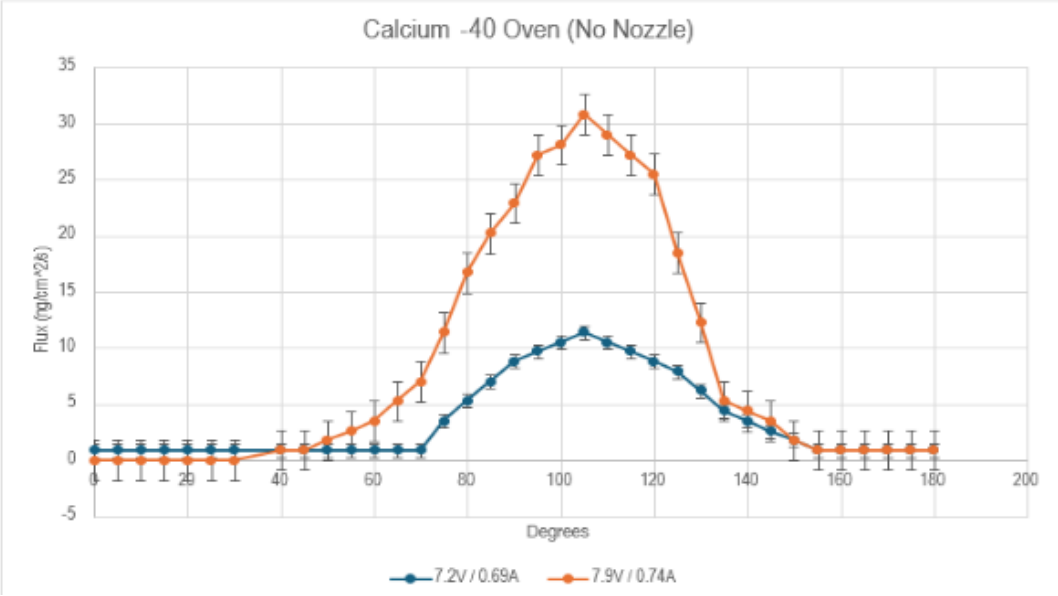

Figure 3.3 Data collected from our Calcium – 40 oven that has no nozzle component on the crucible. The flux distribution is plotted over a solid angle for two distinct temperatures. Data was collected every 5 degrees.

When adding our calcium sample, the pieces were placed opposite the exit of the crucible and were laid next to each other. This was done for a few reasons. First, we needed to make sure our sample would not find its way out of the crucible, as it is not uncommon for this to happen had the sample been placed haphazardly. Secondly, we wanted to have space between our sample and the crucible exit, so as to allow for the neutral vapor to interact with our altered crucible design.

This process was repeated at two separate voltages to see if we can locate some sort of connection between our flux and what the increased power can generate. The method for this did not require replacing anything, as there was more than enough of the sample for us to continue taking data with the same setup. Increasing the power further from here would then give more insight into the capabilities of the oven setup.

Looking further at our Calcium-40 data we find an interesting connection. When increasing the power to the oven, we of course see an increase in neutral flux for all the regions of solid angle with significance. On the fringe ends, we are still met with low and insignificant flux. In saying "insignificant", we are referring to the reality that these particles face. A large portion of these particles will end up being absorbed by the walls of the plasma chamber in the ECR source. Now,

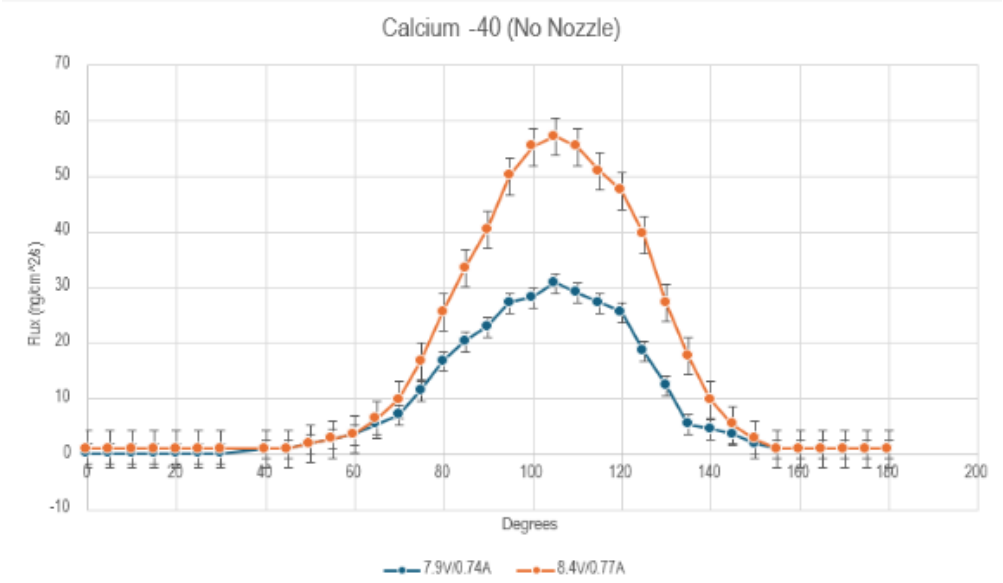

Figure 3.4 Data collected from our Calcium – 40 oven that has no nozzle component on the crucible. The flux distribution is plotted over a solid angle for two distinct temperatures. The 7.9V/0.74A plot is added for reference from the data in Figure 6.

it is very important to record these portions of the solid angle, due to the fact that we need to be optimizing the ovens in a way that makes sure the majority of the neutral flux is being ionized by the plasma.

Returning to the figures for our calcium-40 oven, we are met with an almost Gaussian distribution of neutral flux. Even with an increase in power, the distributions along the solid angle change very little. Consistency is a good thing for these ovens, as when they are turned on in the source, they will run for hours and days without stopping production. The plasma itself, while being heated with RF power, will have an influence on the temperature experienced inside the crucible. In the next section, we will be introduced to a situation where we have to be very careful with how we calibrate our increase in temperature, as it may lead to overproduction in the vacuum chamber. However, for our calcium-40 deposition, we have shown that even with increasing the power applied to the oven we can be assured of consistency with the distribution.

At FRIB, different types of particle oven are utilized depending on the users' needs. So far, a simple resistive oven has been used to generate the necessary flux to record our data. But, it is also very important to consider the differences between different types of ovens that are deployed in the

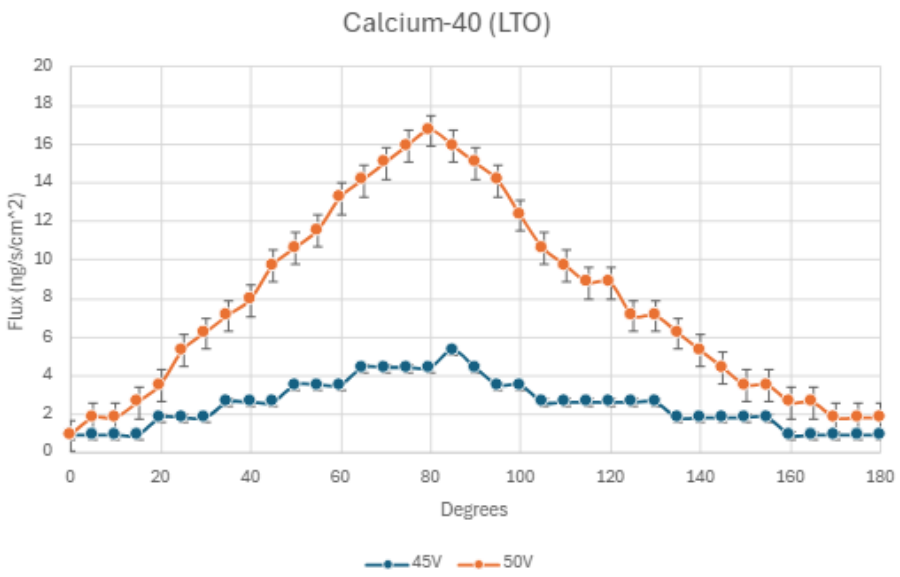

Figure 3.5 Plot of neutral flux distribution for Calcium-40 from a LTO setup.

lab. The current calcium-40 test was repeated with a new type of oven. This Low Temperature Oven (LTO) introduces a cartridge loading system for the crucible that holds the sample. The crucible itself is almost identical to the ones used in the resistive oven setups, but the way it sits within the oven is quite different. **Figure 1.3** is a great example of where the crucible actually sits relative to the opening. The loading of this oven in particular requires the copper head of the oven to be removed, the loaded crucible to be placed inside, and then the head is attached back on with two screws. Due to the design dimensions of the oven itself, we are faced with a situation where the crucible sits in the middle, but the exit hole is off-center. Now, to counteract this, the exit is equipped with multiple tiny holes that try to direct the flux back towards the horizontal center-line of the oven. Now, this type of design has a real influence on our particle flux distribution, which we will discuss more in the next section.

With all aspects of the setup remaining the same, and now introducing an entirely new oven, we are able to witness some drastic differences. First and foremost, when comparing **Figure 3.5** and **Figure 3.3**, you can already see the difference in spreads. The LTO, with its multiple hole nozzle setup as seen in **Figure 1.5**, seems to create a much wider spread of flux. There is much more calcium being accumulated as the crystal sensor moves across the solid angle. For example, even

though the flux values may indicate that the resistive oven is producing more material, this is not exactly the case. Over the period of data collection, the LTO had recorded much more mass than compared to the resistive oven setup. The maximum flux of the resistive oven is higher than the flux seen by the LTO, which is important to consider going forward.

Although the oven and the crystal sensor are aligned, the geometry of the oven exit is noticeable in the data. This exemplifies a very important aspect of the process in which we set these ovens up. You must take into account the way that the oven exit and nozzle will be pointing when inside the vacuum chamber. The crystal sensor is on a rotating flange by design for data collection, but the oven will not usually be on that type of flange fitting. This means, that once the oven is set there is no way to adjust that type of orientation unless the vacuum is reset. Careful consideration must be taken, especially with these Low Temperature Ovens.

3.3 Zinc Oven Test

The process for generating Zinc metal deposition from a resistive oven follows the same procedures as were covered in the previous section. There is a bit of nuance to tuning the oven power for the Zinc oven, which is worth covering. To start, we wanted to continue using the crucible setup with no oven nozzle, as it gave us very good results with our calcium ovens.

Zinc proved to be a tricky material to accurately deposit. This type of material can be easily over-produced and can cause issues within vacuum. Zinc has a relatively low vaporization point compared to that of calcium, so being able to produce enough power would not be an issue. The issue involved the lack of temperature control and leaving the power to the oven high enough for too long. The oven, when set to 5V, produced too much zinc inside the vacuum chamber that it had covered the see-through glass flange. When the microbalance was turned on to collect data, it was unable to accurately display any stable flux from the oven. It was now apparent that the crystal would have to be replaced and the power to the oven would have to be closely monitored. The crystal holder had the zinc removed and the crystal itself was replaced with a new crystal that had more life expectancy.

Once the crystal was replaced, along with the oven as well, testing could continue. The

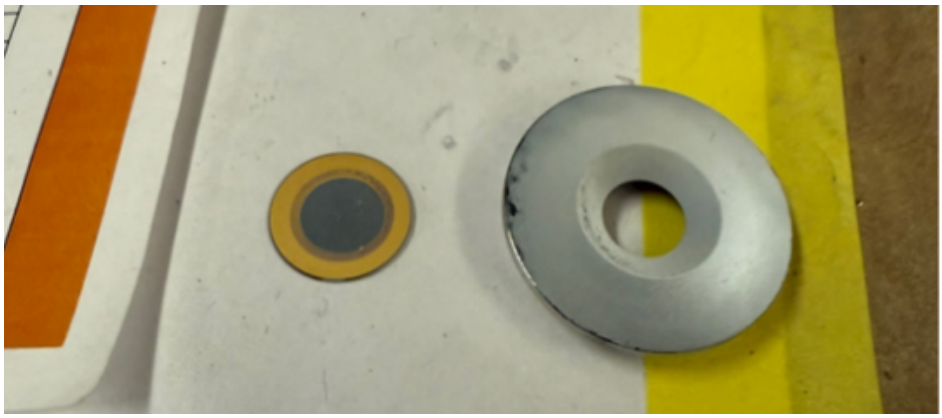

Figure 3.6 On the left is a gold-plated quartz crystal coated in excess amounts of zinc. The relative crystal life was depleted significantly due to this incident. On the right is the crystal holder made of stainless steel and is also coated in zinc.

Time	V/A	Mass (nanograms)
4:05 pm	3.5 V/ 0.40 A	0.1653
4:45 pm	3.5V/ 0.39 A	0.3128
5:31 pm	3.5 V/ 0.39 A	1.519

Table 3.1 Zinc resistive oven with mass values recorded at 90 degrees solid angle. Time is recorded to show the procession of the accumulation of mass detected by the microbalance.

microbalance was now going to be used to confirm the correct voltage and amperage we would want to record our flux distribution data at. This was a trivial process that required the oven to be set at a specific voltage, and then slowly increased over time. This was to pinpoint exactly where we should be recording our data.

The oven was slowly increased over time to reach 3.5V. This was the minimum voltage at which we were able to witness significant mass accumulation on the crystal surface. For reference, in **Table 3.1** we have a situation where the flux is very low and somewhat insignificant, but over a short period of time we are collecting mass as the oven continues to heat up. The increase in temperature of the crucible is denoted by the decrease in current on the power supply.

This specific voltage will serve as a very good starting point for data collection. And from here we can revisit the higher voltage that had clouded our previous experiment, which was around 5V. At this specific voltage, we witnessed extreme levels of flux from the oven. The max flux, which was recorded at 90 degrees solid angle, was at most 255 ng/s/cm^2 and climbing. Even at 0 degrees

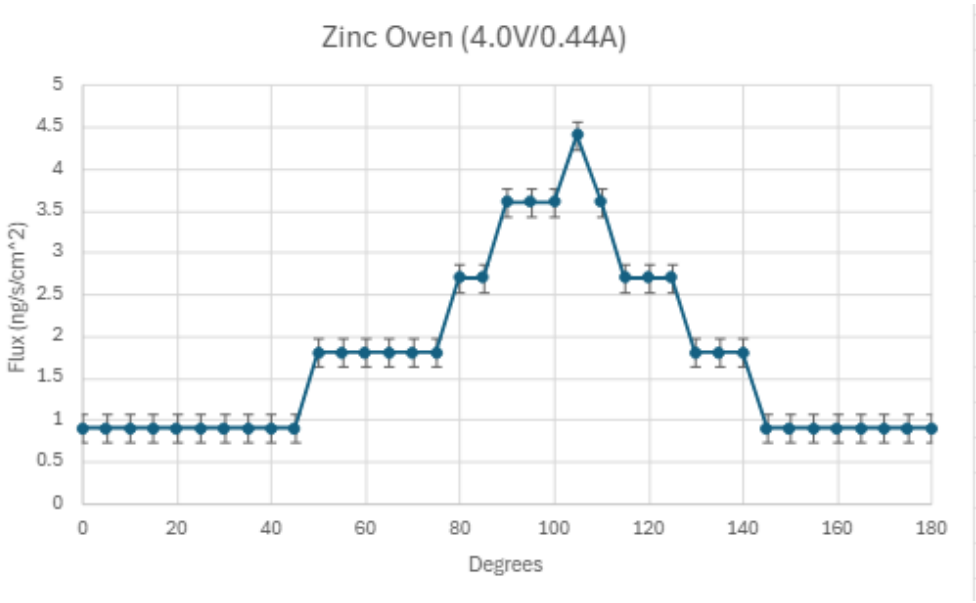

Figure 3.7 Plot of neutral flux distribution for the resistive oven loaded with Zinc. The crucible itself has no nozzle on it.

of solid angle, we were recording a flux of 8 ng/s/cm^2. This type of temperature in the crucible was quite unstable, and it was necessary to find a more suitable voltage range. This type of extreme flux could exhaust the sample that is loaded in the crucible and inhibit the ability to continue testing. Somewhere between 3.5V and 5V would be the range at which we will find stable deposition.

The shape of the distribution in **Figure 3.6** is very reminiscent of our first test with the calcium oven that included a nozzle. The lower temperature could mean a few things in regards to the particles interacting within the crucible. First of all, there is less thermal energy being transmitted to the material, which could result in fewer collisions between the species. Less collisions could in turn amount to less spread as the particles are exiting the crucible. Going further, when we compare it with our data taken at a higher voltage, we see a much sharper peak. This is seen in **Figure 3.7**, where the oven has only increased by 0.4 V. A significant increase in flux, with only a small increase in power demonstrates the necessity for accurate temperature control, especially if you want to employ the use of a crucible design that does nothing to limit neutral flux production.

The testing of the zinc in our resistive oven setup showed just how volatile our production could potentially be. Even with the oven being tuned to a much lower temperature compared to our prior

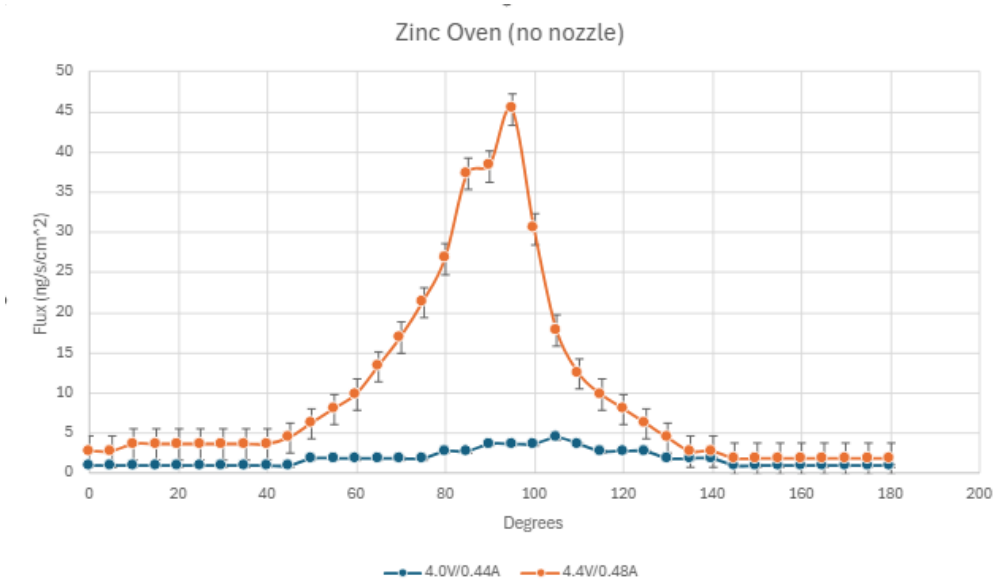

Figure 3.8 Plot of neutral flux distribution for Zinc from a resistive oven setup. The blue plot is the same as Figure 9, added for reference.

tests with the calcium-40, we had to maintain careful consideration of the output from the oven. When we look closer at the figures for our flux distributions, we realize that the peaks of the plot seem to shift as well. This is an interesting observation that could be due to the fact there is no nozzle on the crucible. Over time, as the sample starts to be vaporized, it is common for the solid pieces to move around. The peak seems to shift about 10 degrees in solid angle, and this instability of the vapor production, is most likely brought about by the lack of nozzle on the crucible. No nozzle on the crucible essentially allows the vapor to flow without being forced to collimate towards the smaller exit. This type of oven setup and design will leave you with drastically more flux leaving the oven, but you could potentially be sacrificing certain focusing strengths necessary for accurate capture into the plasma.

CHAPTER 4

DISCUSSION AND ANALYSIS

4.1 Vapor Pressure Curves

Vapor pressure curves are a facet of physics which multiple other studies can rely on. These curves tell a lot about the specific material being used, and the physics behind certain observable measurements. In this study, we have empirical data that points to a drastic increase in flux for both calcium and zinc. This leads us, as all scientists should do, to question our own findings and look for other resources to corroborate our findings. Studies done on vapor pressure curves have long been used to reinforce knowledge within the material sciences [6]. For our understanding, we will be looking at a specific region on the vapor pressure curve for Zinc. We have a very good idea of where we are on the curve, because we know the approximate temperature due to us knowing exactly what voltage and resistance we are at when the data was recorded. From here we can follow the chart from **Figure 4.1** to give us our theoretical vapor pressures. After we have this information, we can use the Ideal Gas Law which is shown by Equation 2.

$$PV = nRT \quad (2)$$

The variables, P and T, represent the values acquired from our vapor pressure curve in **Figure 4.1**. And, it should also be noted these vapor pressure values were validated with a cross reference from a recent published study out of Pennsylvania State University [8]. The volume for our hypothetical crucible is 1 cubic centimeter, which is almost the same volume as the crucible used. R is constant and we will be analyzing our results for the number of particles, n. After solving for n, we use the density of Zinc and Avogadro's number to put the answer in perspective of the increase we have observed in the data. **Table 4.1** has our calculated values for both "n" results representing T1 and T2. From our theoretical calculations, we should expect to see an increase in the number of particles in the range of somewhere between 8-10 times. And, if we observe what kind of increase we have at the same solid angle for where the maximum amount of flux is observed, it will be within the range of this estimate.

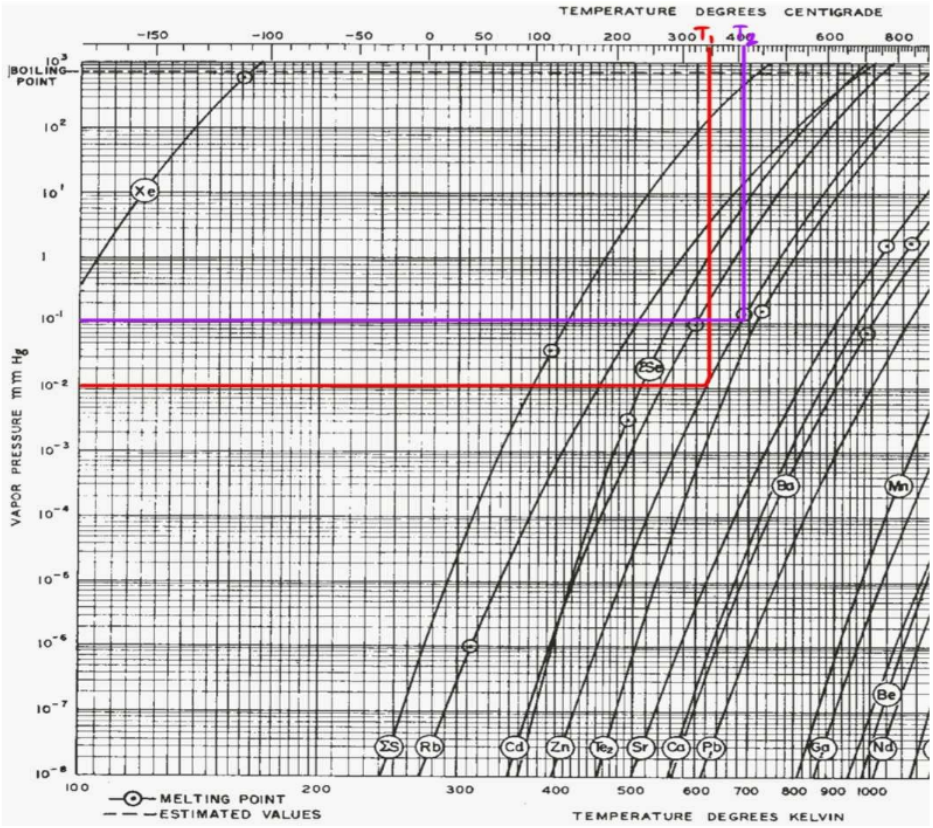

Figure 4.1 Vapor pressure curve for Zinc. The purple and red annotations correspond to the two distinct voltages from Figure 3.8 on the Zinc particle distribution.

T_1 = 350 C	T_2 = 420 C	Increase
4.4 ng/s/cm^2	45.3 ng/s/cm^2	929.453%
0.1262 ng	1.135 ng	799.366%

Table 4.1 The T1 and T2 denote the specific temperatures highlighted in Figure 9. These temperatures are calculated from the resistance change in the wire of the oven. The second row is the experimental flux values taken from the peak solid angle and compared with theoretical values derived from our vapor pressure curve.

These types of theoretical calculations can be very helpful in showcasing the effectiveness of our microbalance. Going forward, the capabilities of the quartz microbalance system may require different uses depending on the lab's goals and the needs of the department. The sheer number of applications of this type of technology may end up going beyond that of improving and optimizing the particle ovens used in the ion sources.

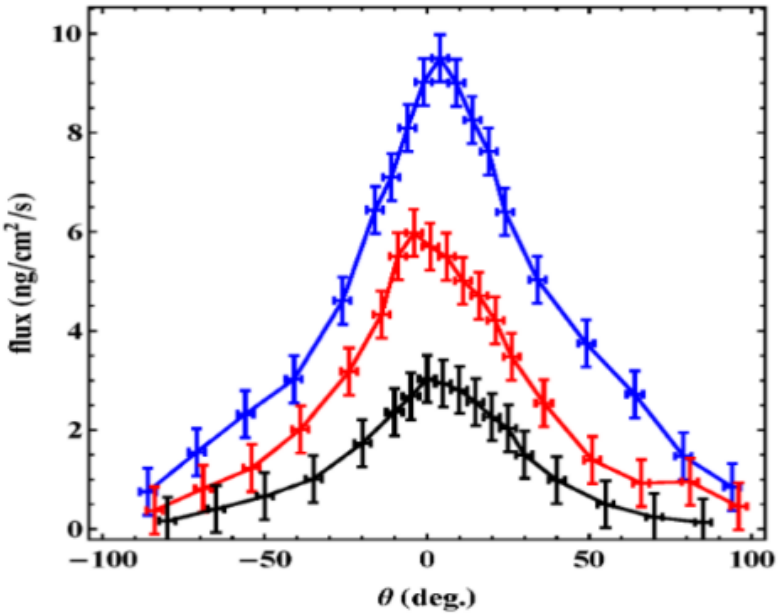

Figure 4. Experimental flux emitted from the oven as a function of the angle θ for calcium. The black, red and blue plots are respectively measured at the temperatures 850, 875, 900°K.

Figure 4.2 Plot and figure description are from [7]

4.2 Particle Flux Distribution and Observations

When considering the validity of our data collection, it is important to cross-reference our results with other peer reviewed papers. This is exactly what we have done in reference to **Figure 4.2**, which is noted as being taken from an aforementioned paper [7]. Looking at **Figure 3.2** you can already notice a few similarities. For instance, the shape of the distributed plot seems to be quite similar. The peak for the flux in both figures is around 10 ng/s/cm^2 as well. It should also be noted that when calculated, the temperatures produced from the resistive oven are within the same range as the temperatures highlighted in **Figure 4.2**.

The crucible design deployed also plays a major role in what kind of material flux we are seeing. For instance, the Calcium-40 tests for a crucible with a nozzle, and the ones with no nozzle, displayed major differences. The increase in flux was, as mentioned before, quite significant. But looking beyond that, we are witnessing a situation where the crucible with the nozzle does a much better job at directing its flux and collimating it towards the center of the solid angle. While for our tests with the crucible that has no nozzle, the spread of the flux is not as controlled. It was

mentioned in a previous section that the fate for a majority of the flux will include being absorbed by the chamber walls. While we do appreciate the increase in flux, it is also important to consider whether or not this flux meaningfully impacts our production of ions. Overproduction can lead to contamination within the source. This was very apparent with the zinc oven test, and is something to consider when looking to potentially utilize an oven crucible with no nozzle.

Let's consider a numerical outlook for some of the data we have collected from the various ovens. The word "spread" has been used quite a bit to characterize the effects that the different types of oven and crucible designs have had on the neutral flux data. A very simple calculation of standard deviation may paint a more accurate picture of what the results actually mean. For example, in **Figure 3.3** the data corresponding to 7.2V/0.69A has a standard deviation of 3.63. Meanwhile, the data from the 7.9V/0.74A plot gives us a standard deviation of 10.54. This is a dramatic increase in standard deviation, which indicates just how much more of a spread we are witnessing from the mean. Doing this again for the data in **Figure 3.4** reveals a standard deviation of 19.89 for the 8.4V/0.77A plot. Now, these numbers are fairly arbitrary on their own, but when we compare the change in standard deviation with the data from our calcium-40 LTO, we can start to contextualize the meaning behind these numbers a little better. For the standard deviation of the 45V data from **Figure 3.5**, it is calculated to be 1.25. And the 50V data generates a standard deviation of just 4.87. Increasing the temperature of a crucible that has no nozzle seems to significantly increase the spread of the flux that was recorded by the microbalance, according to this data. Meanwhile, for our LTO setup with the same species loaded in the crucible, increasing the temperature will still generate more spread, but it will not be as significant as the resistive oven setup. This type of data speaks to the stability of the LTO when in comparison to our resistive oven. Using this type of calculation could bring an important contextualization to future developments of particle ovens.

This research and methodology also gives us the ability to influence future oven and crucible designs. Ideally, you would want to design a crucible that has little to no absorption of the metal sample, and collimates all the flux to be expelled in one singular direction. One way that could help do this is to potentially make the total volume of the crucible larger. If the volume of the

crucible were to be made larger, we could potentially see more of the particles inside interacting with each other instead of the walls of the crucible. For example, the sticking time between two particles of calcium is much shorter than the sticking time of calcium and molybdenum [7]. More collisions that occur within the crucible for particles interacting with each other,and not the walls of the crucible, could potentially see an increase in flux exiting the oven. And to go a step further, implementing and testing different types of exits and nozzle shapes could lead to better collimation. Being able to accurately focus the spread of the flux will not only limit contamination inside the source, but could potentially save money for the department as well. All of these new developments can now be tested beforehand with the use of the quartz microbalance system, without having to be ran in the sources.

Stability is a very important concept when it comes to the science of accelerator systems. In light of this, we should consider how the stability of each particle oven can have an effect on the production inside the source. The resistive oven, when compared with a cartridge style oven like the LTO design, is much more susceptible to temperature change. The crucible used for the LTO sits inside a metal head attached to the end of the oven, as seen in **1.6**. This type of design works to insulate the crucible, which is important when considering the proximity of the oven to the hot plasma.

CHAPTER 5

CONCLUSION

Optimizing an Electron Cyclotron Resonance ion source is no easy task. It takes an entire department of skilled engineers and physicists to maintain these machines, and run them effectively. The development of a process in which we can optimize an important piece of mission critical equipment is quite substantial. This will open up new pathways for future particle oven developments, along with being able to keep up with the increased demand for higher beam power. There will potentially come a time where the abilities of the source could outperform the capabilities of the ovens being utilized. It is important, as with all aspects of accelerator systems, to not overlook and neglect certain systems just because they work fine now. Improving these types of things will only raise the floor of all the systems that go into making an effective and powerful particle accelerator system.

The methodology showcased within this thesis will help the scientists and engineers within the department to continue developing solutions, all without impeding the production schedule of the lab. This will in turn, create a better product for users, and streamline progress in the fields of research and development.

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