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DESIGNING A COMPLEX SUBSTRATE HEATER FOR THIN-FILM DEPOSITION IN HIGH
VACUUM

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DESIGNING A COMPLEX SUBSTRATE HEATER FOR THIN-FILM DEPOSITION IN HIGH
VACUUM

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SCHOOL OF AEROSPACE AND MECHANICAL ENGINEERING

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Abstract

Thin films are a ubiquitous resource within condensed matter research at nanoscopic scale. This thesis outlines a custom substrate heater for use in physical vapor deposition of thin films for laboratory use. This heater sandwiches the substrate between two ceramic heat sources to reduce radiative heat loss and more easily bring it to thermal equilibrium.

1 Introduction

The Bumm research group within the Homer L. Dodge Department of Physics and Astronomy studies condensed matter physics, specifically surface physics at the nanometer scale. This research relies heavily on being able to study matter at a molecular scale, and thus requires unique, highly specialized scientific equipment and manufactured components. For our group, this includes scanning tunneling microscopes, supercomputers, and thin films. Recent advancements in supercomputing and microscopy have made direct measure of nanoscale properties in thin films achievable, and thus a priority for the Bumm research group's work. Subsequently this has made thin films a vital resource. Thin films are nanoscopically thin materials made up of finely controlled layers of deposited matter. Thin films are a useful research subject for the study of surface physics, which is a useful science for the further miniaturization of transistors. The breakthrough which led to the invention of the metal-oxide-semiconductor field-effect transistor, or MOSFET, was the controlled growth of thin layers of silicon oxide on silicon wafers using thermal oxidation and the surface physics which governed this reaction. The MOSFET lead directly to the microchip revolution and then the digital revolution [1]. Less than a decade after the MOSFET the first thin-film transistor was created, which was eventually used and made essential in the production of LCD screens which are now a part of daily life [2]. Clearly, work and research at this scale has broad and useful applications.

Many suppliers produce thin films for research use, but the ability to produce bespoke films on-site can be more cost effective while also allowing for unique film compositions that suppliers can't provide. Some materials, like silver, would tarnish in the time between commercial manufacture and delivery onsite. This is clearly undesirable if one is wishing to study a film made of silver, rather than silver sulfide. The reason many labs choose to buy films instead of making them onsite is that the upfront cost of the equipment required can be quite great.

Of the ways to produce thin films, thermal deposition within a high, or ultra-high, vacuum is very simple, assuming one can produce such an extreme environment. These levels of vacuum

Degree of Vacuum		Pressure Range (Pa)	
Low	10^5	$> P >$	3.3×10^3
Medium	3.3×10^3	$> P >$	10^{-1}
High	10^{-1}	$> P >$	10^{-4}
Very High	10^{-4}	$> P >$	10^{-7}
Ultrahigh	10^{-7}	$> P >$	10^{-10}
Extreme ultrahigh	10^{-10}	$> P$	

Table 1: A table delimiting what degree of vacuum corresponds to what pressure range

are not universally defined, but are generally accepted as falling within the ranges presented by Table 1. In fact, the process is so simple that even Edison was able to perform various depositions within vacuum, filing patents in 1880 and utilizing gold deposition in a process of strengthening phonograph masters for mass reproduction from 1901 to 1921 [7]. The ubiquity of this process partially contributed to the impetus for this project, as a Varian 3118 vacuum chamber was inherited by the Bumm group and thus provided the base, and most expensive component, of the necessary equipment. Despite being manufactured decades ago, it was fully compliant with ConFlat® components which are still widely available commercially, offering a starting point to design a substrate heater for thin film manufacture.

While commercial solutions do exist for substrate heating, creating a fully custom setup offers opportunities to prioritize the expected use-case of the heater. Examples include dramatically insulating the heater to better retain radiated heat, making the substrate easily accessible, and integrating multiple heating sources to ensure uniformly distributed heat in the substrate. With these benefits in mind, a design was pursued to combine as many of these priorities as deemed feasible, to produce an effective and useful substrate heater.

2 Design Outline

The needs of this system, including its intended and potential applications, were the first considerations of this design. Additional consideration was given to the intrinsic limitations which come with vacuum applications, and how the behavior of substances can dramatically change under vacuum.

Thermal evaporation is an effective physical vapor deposition (PVD) process specifically because of the benefits a vacuum environment can provide. The vacuum maximizes the rate of evaporation by keeping the surrounding environment unsaturated and the vapor pressure the sole source of pressure. All of these considerations are based off of most thin films needing to be metallic, and the incredible energy required to liquefy metals.

Thermal evaporation and deposition requires both a source to evaporate and a substrate to adsorb the gaseous particles. This project dealt with the substrate, more specifically the assembly required to maintain the highest quality adsorptive environment, one which kept all areas of the substrate uniformly heated, and thus predisposed to even, predictably thickening thin film production. This system also needed to heat efficiently, as the preferred temperature range for deposition frequently reaches 1000°C [3]. At this temperature range it is very easy to incidentally emit enough heat to damage or even crack the enclosing glass bell jar. Keeping in mind that even a low vacuum environment severely reduces convective heat transfer, it's easy to see how unaccounted for heat poses a serious risk to the operative capacity of the setup.

2.1 Water-Cooled Walls

Almost all of the system's components are encompassed by the thick copper walls which make up the outside of the 'box'. The high thermal conductance of the copper makes it ideal as a sink material to remove unaccounted for heat in the system. Additionally, copper's machinability means that more complicated manufacturing operations can be employed, and special capabilities can be utilized in the final design.

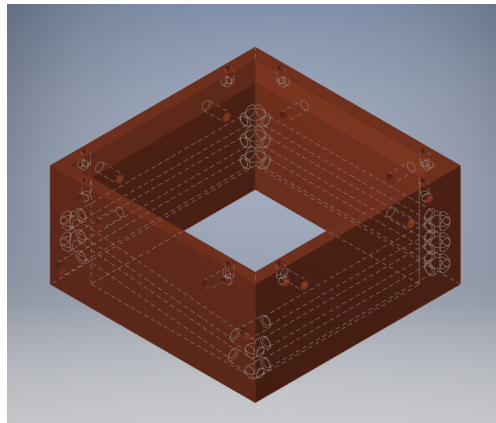


Figure 1: The 3D model of the heater's walls, with transparency enabled such that the water-cooling lines are visible

This design employs a significant amount of cooling through the use of stepped waterlines, made by through holes drilled in the center-line of each wall with plugs affixed to the entrance and exit holes. This water removes the escaped heat of the ceramic heaters, preventing that heat from escaping into the rest of the chamber. This is made necessary by the balancing act required by thermal deposition within a vacuum. While the substrate and the source need to be heated, sometimes to temperatures exceeding 1000°C , heat elsewhere within the chamber can pose significant obstacles.

Off-gassing is the process of a solid releasing gas trapped within it, and poses a constant maintenance challenge within vacuum environments. Atmospheric pressure can provide an equilibrium of force to trap the gas, but its absence within the vacuum leads to an initial release of gas which is handled by the roughing pump. But the application of heat allows for further off-

gassing, as Gay-Lussac's law can tell you the pressure of a gas, even that trapped within solid bodies, is proportional to temperature, so the application of heat results in an increased pressure of the trapped gasses and thus further release, maintaining equilibrium with the vacuum. The reason the equipment which provides the vacuum cannot simply remove all incidental off-gassing is that higher levels of vacuum require more sophisticated vacuums, which adsorb gaseous particles, rather than simply removing them to the chamber's exterior. Thus, they can become saturated with adsorbed gas during operation, and remaining free gas will continue unabated in the chamber. For this reason it's important to apply heat at the roughing stage, before any deposition takes place, to maximize the off-gas removed by the comparatively bottomless roughing pump. But because it's impossible to remove all of this gas, it's essential to minimize any unfocused heat within the chamber, in turn minimizing the off-gassing. An additional danger heat poses for this setup is the potential to thermally shock and shatter the glass bell jar which encompasses the top of the chamber. As one might imagine, the chamber's integrity is a foremost concern when working with a vacuum chamber.

A discussion of the methods used to calculate the needs of this system is found in section 4.2, and the CFD modeling techniques used to validate this design is the subject of chapter 5.

2.2 Lid

While vacuum chambers have an immense number of applications, this one will only be used for thin film depositions for the foreseeable future. This means that it needs to be as simple and quick as possible to replace the substrate, once the desired film has been deposited and collected. This system addresses that need by mounting the substrate holder to the lid of the setup, so that only one face of the 'box' need to be removed to access the substrate holder, and it's the top-most face to boot. The lid contains a slot within the lip which is sized to ease prying the lid off the box's top. The lid also features a hole in its center, to accommodate the actuated heat-sink, which is discussed in the following section.

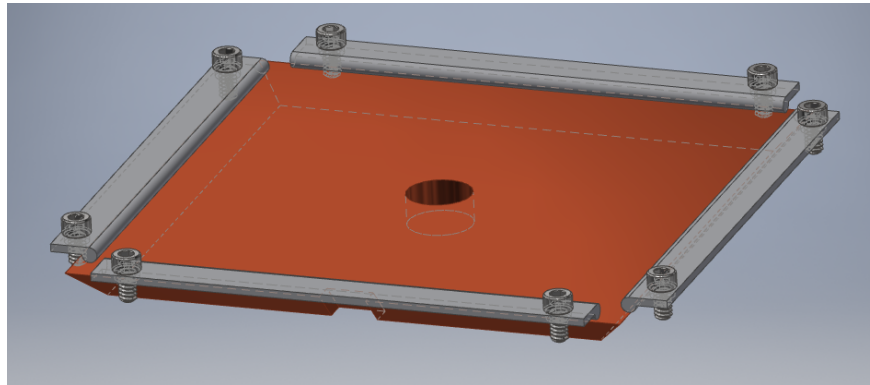


Figure 2: The heater's lid, isolated from the rest of the CAD assembly

And on the underside of this lid, seen in figure 3, there are layers of tantalum for insulation, and the mounting hardware for the alumina block. The alumina block will have coils of tungsten-rhenium through it, then electrified, producing heat through its internal resistance. The desired substrate will be clipped onto the block, and the block will be secured through bent wires, to reduce heat wasted through conduction into the rest of the assembly.

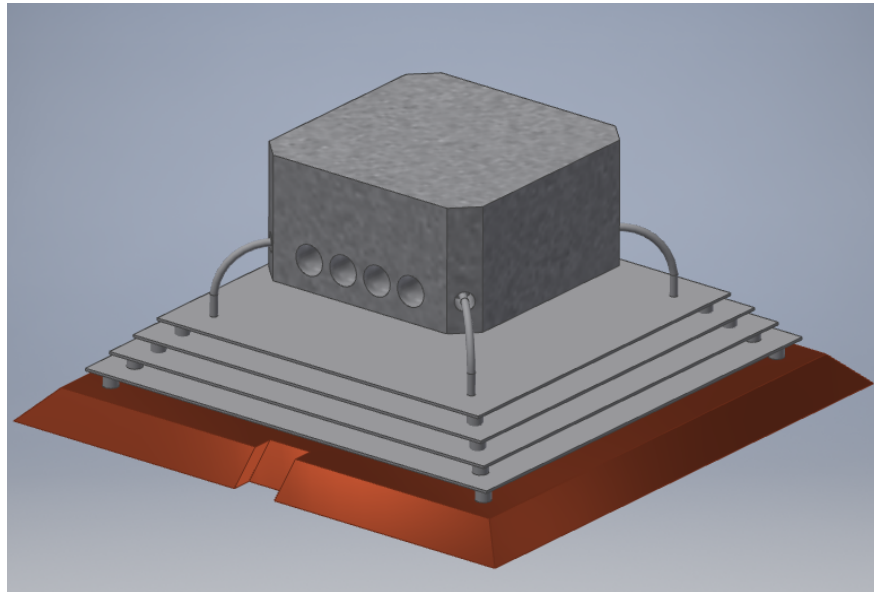


Figure 3: The underside of the heater's lid, isolated from the rest of the CAD assembly

2.3 Actuated Heat-Sink

Every aspect of this design focuses on improving the usability for thin film deposition, and that includes improving the duty cycle of the setup. The heat sink's role is to expunge lingering heat once a deposition has completed. Without this component, one would have to wait up to several hours for all of the heat to safely dissipate from the heater's interior, due to the combination of its insulating properties and the lack of convective heat transfer. This heat sink gives the operator a shortcut, offering the heat a direct conductive path from the alumina to the copper exterior, and better yet the heat wicking water lines. But actually actuating this heat sink offered a challenge.

In atmosphere, oftentimes what appears to be conductive heat transfer is heavily supplemented by the convective layer. This means that, while two bodies may appear to be touching, the surface area in contact may be very small. In this case, there's an apparent need to align two axes in three-dimensional space to achieve the highest quality contact. This can be difficult when manipulating equipment outside of a vacuum chamber, never the less within one. So to compensate, our actuation scheme takes advantage of the indiscriminate compressive force of a spring. This force maximizes the conductive area, and thus the heat conducted out of the block.

Much like an active high switch, when the spring isn't displaced it presses the solid cylinder of copper, the sink, against the alumina, the source. By actuating this setup with a cord, it's difficult to incidentally apply too much force to the alumina block and crack it. It also simplifies the process of uncoupling these actuation pieces when the lid needs to be removed for accessing the substrate.

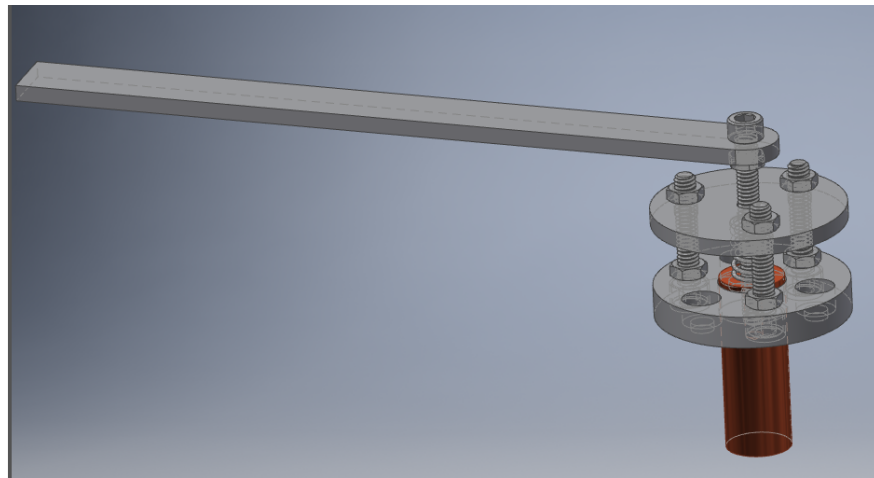


Figure 4: The model for the assembly of the actuated heat-sink. The heat-sink itself can be seen as the copper cylinder at the bottom of the image.

2.4 Shutter-Cart

The traditional shutter of vacuum setups is the basis of a surprising number of components. This is at least partially because by its simplest definition, a surface which can be voluntarily moved in-and-out of the way of the deposition source, a shutter can accomplish the vast majority of desired operations. The simplest shutters allow technicians to precisely control the amount and quality of adsorbed material, by preventing deposition until a steady flow is achieved and only allowing deposition for the desired amount of time, thus controlling the number of layers. In a similar respect, by placing an electrified quartz crystal on a shutter one can measure the deposited material by the damping of the quartz's vibrations as its mass increases with deposited material. By intermittently shuttering the deposition source one can slow the deposition rate proportionally to the shutter's uncovered portion.

The bottom shutter is essentially a mirrored and compressed version of the system lying above it. This is another utility-minded choice. A totally passive shutter would only maintain a barrier between the evaporating source and the adsorbing substrate. This barrier allows the operator to finely control the amount of deposited material in the thin film. However, it does nothing else. Our shutter has greater purpose behind its design.

Heat transfer within a vacuum is complicated by the inherent absence of a conductive fluid. In atmospheric conditions, one might think of a pan set upon a hot-plate as an example of thermal conduction. In reality, much of that transfer was accounted for by the incredibly thin layer of air maintained between the pan and the hot-plate, which can be presumed to be in an imperfect degree of contact due to minor irregularities in each surface. This simplification is generally unaddressed in daily life, with limited exceptions such as the use of thermally conductive paste replacing the layer of air in the interface between computer components and their respective cooling accessories. Such pastes are an inappropriate solution for this setup, as their off-gassing and effective temperature range are both nonstarters.

Instead of relying on a layer of fluid to facilitate effective heat transfer, this design emphasizes

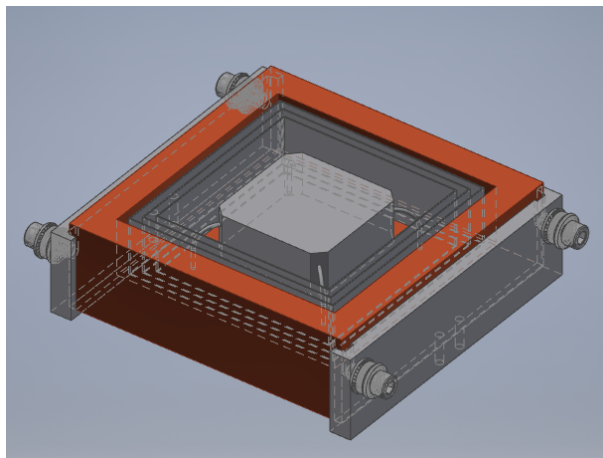


Figure 5: The shutter-cart, as modeled. At each of the four corners there's a bearing which is fastened such that it can freely rotate, and act as the "wheels" of the shutter-cart.

the radiative application of heat to the substrate. While the substrate is affixed to the top ceramic tightly to provide some conductive transfer, the Shutter-Cart's heater's radiative heat effectively doubles that which's provided by the above heater. In this way the substrate is hopefully brought to the desired temperature, and an equilibrium, in a more uniform manner, which provides a more desirable surface for the intended deposition to adsorb onto.

Starting from the outside and working in, the copper walls function identically to those of the top, allowing a reservoir for remnants of escaping heat. Then layers of tantalum insulate the heater from radiative heat losses, reflecting that heat back at the heater to help it build in temperature. Finally a duplicate alumina heating block radiates heat directly at the substrate above. In this way the substrate is heated from both of its sides, and approaches its desired heat sooner as a result. Of course, all of these components contribute to the overall weight of this piece of the system, but that's why the rails have been made so accommodating.

2.5 Rails

The complexities of this heater design bely a much greater effectiveness of the system as a whole, but the benefits do come at significant costs. One of the largest design sacrifices made was that of weight. While an ordinary setup's shutter could weigh mere ounces, ours weighs nearly 7 pounds, over 3 kilograms. This is a trivial amount when manipulated outside the chamber, but when hung above thousands of dollars of equipment within a sealed container replicating the conditions of space, it's another proposition entirely. Therefore, instead of merely mounting the shutter on a rotary feedthrough, the entire shutter is mounted to a cart, which sits upon two rails which are mounted to the system's overall support frame, which is affixed to four separate mounting rods inside the chamber. The overall support frame also incorporates shields which prevent the shutter's heat from going straight into the bell jar when displaced from under the top heater.

In designing this way, the shutter will always be supported, no matter what systems might break. Additionally, this strictly dictates the motion of the shutter, given the operator precise and rapid control along the length of track.

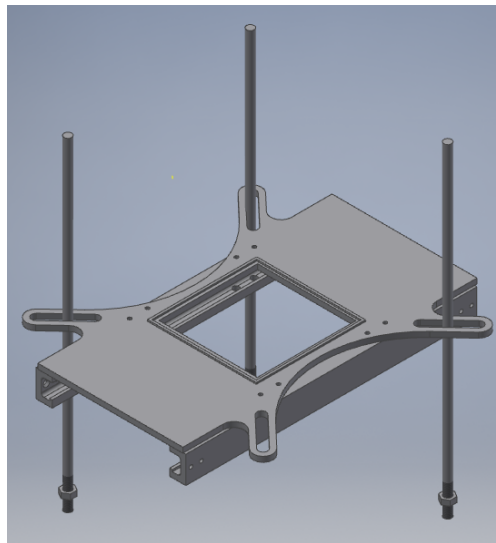


Figure 6: The rails and their associated structures, the plate which the heater rests upon and the posts which carry the entire structure, with two posts omitted for visual clarity.

2.6 Actuation

Actuation within the chamber became a huge cause of concern as the project went on. At least two components of the heater assembly needed to be movable from the outside. Each proposed solution came with benefits and drawbacks which needed to be compared in order to decide upon an agreeable solution. While certain off the shelf solutions addressed these needs, their prices increase exorbitantly with their capabilities. An effort has been made to reuse existing equipment, so working within their bounds is a fiscally minded choice.

When considering actuation options it's important to consider that many solutions for transferring bases of motion in atmosphere are inappropriate in a vacuum. The friction of imprecise gears can cause particulate spikes, heat can be conducted or stored in troubling ways, pneumatic choices are completely off the table, lubricants and other coatings are often sources of significant off-gassing, and standard definitions of close or loose fits may rely on the presence of atmosphere to correctly apply.

Balancing the priorities of a given solution and their accompanying drawbacks is a priority in this project. Naturally, proposed solutions need to

cart actuation

The challenge of actuating the cart rested on balancing the various desirable features of any given actuation solution.

heat sink actuation

3 Mathematical Analysis

3.1 Radiative Heat Transfer Calculations

This step of the thermal evaporator design process is to create an effective substrate heater. The heater's effectiveness is to be evaluated based on how evenly it provides a 1000° C face of an alumina block, with dimensions of approximately 3 by 3 inches, which our substrate will rest upon; and how efficiently it uses its supply power to reach this temperature.

Simplified analysis began with an assumed shape of an alumina block, 3x3x1 in, with a surface area of 30 in² (0.0193548 m²), heated to a steady-state (isothermal) of 1000° C (1273.15 K). We will initially treat this shape as a sphere of equivalent area.

Using this first rough assumption, we can calculate the approximate power radiated from an akin blackbody using the Stefan-Boltzmann law (1).

$$P = A\sigma T^4 \quad (1)$$

Where

$$\sigma = \frac{2\pi^5 k^4}{15c^2 h^3} = 5.670373 \times 10^{-8} \text{ Wm}^{-2}\text{K}^{-4},$$

$P \equiv \text{Power Radiated, W}$
 $A \equiv \text{Surface Area, m}^2$
 $T \equiv \text{Absolute Temperature, K}$

This initial estimate offers

$$P \cong 2889 \text{ W}$$

This heat loss indicates that we would need to supply that level of wattage to maintain the desired temperature of the block. As this is 4 times the power required by a reasonably priced microwave, this is not an ideal approximation. It also does not account for losses due to conduction, and the efficiency (or inefficiency) of the desired heating method, tungsten-rhenium wire, which radiates heat when amperage is applied, due to its electrical resistance.

The first refinement to make is to assume an isothermal environmental temperature, that being the temperature of the chamber walls. Starting with 25° C, standard ambient temperature,

$$P = A\sigma \left(T_{\text{body}}^4 - T_{\text{surroundings}}^4 \right). \quad (2)$$

This only reduces the required heat to 2,875 Watts.

Treating the heater and the chamber walls as objects with arbitrary geometry, instead of formless concepts, can help narrow down the actual expected wattage. Using what's called the shape factor, or view factor, F_{i-j} , for bodies i and j is the first step in understanding the importance of the physicality of the surfaces.

These are only functions of geometry, and act as coefficients to the terms in the black body emission calculations (3).

$$\dot{Q}_{1-2} = A_1 F_{1-2} (E_{b1} - E_{b2}), \quad E_{bi} = \sigma T_{bi}^4 \quad (3)$$

In the case of a body fully enclosed by the surface of another body, like our approximation as it currently stands, or a pair of concentric circles, the view factor is $F_{1-2} = 1$, as all emitted radiation is captured by the enclosing surface. This level of approximation does not account for what fraction of emitted radiation reaches the surface which emits it, F_{i-i} . For the case of two infinite parallel planes, this is zero. But for 2 concentric spheres, per our approximation, this is a nonzero fraction. This discrepancy will be accounted for later.

The next assumption is that layers of tantalum foil will be used to reduce the radiative losses of the block, as this is a standard insulating material in alike applications. In conduction analysis there's a concept of thermal resistance, in analogy to electrical resistance, to calculate the reduction in heat transfer in a system due to the presence of a given material. Though this approximation is far from accounting for whatever conductive heat transfer may occur, this technique can still be used to better calculate the radiative heat losses. Before accounting for the emissivity constants, ϵ , and treating the block as being fully shrouded by its foil, then again shrouded by the

chamber walls, it's obvious from the zeroth law of thermodynamics, that of equilibrium, that the tantalum foil must reach a temperature between that of the surroundings' sink, and the block's source. In this approximation (before accounting for the emissivity values), every N layer of foil reduces the heat losses by a coefficient of $\frac{1}{1+N}$, with such a limit that 'infinite' layers fully eliminate heat transfer, but being impossible in that 'infinite layers' are simple a continuous solid, and thus being very effective at conducting heat within itself.

$$P = \frac{1}{1+N} A \sigma T^4 \quad (4)$$

With this coefficient in mind it's trivial to determine the new radiative heat losses at an N layer of insulating foil. However, once one accounts for the emissivity values, the foil's insulating efficacy is reduced.

Emissivities are a fundamental component of our approximation. These coefficients, represented with ϵ , work alike to the view factor, except they're independent of the final geometry. They're merely functions of what materials each body is made of, and sometimes what temperature as well. This step of the analysis will begin with the high-end approximate values for these, so that the approximation favors overcompensating for the system's requisite power.

Pulling from the table provided here [http://www-eng.lbl.gov/dw/projects/DW4229_LHC_detector_analysis/calculations/emissivity2.pdf] we have a number of applicable emissivities.

$$\epsilon_{\text{Al}_2\text{O}_3} = 0.42$$

$$\epsilon_{\text{Ta}} = 0.26$$

$$\epsilon_{\text{Cu}} = 0.88$$

For our two body, heater and surroundings, case, this modifies equation 1 such:

$$P = \epsilon' \sigma A (T_{\text{body}}^4 - T_{\text{surroundings}}^4) = 1,142 \text{ W}, \quad \epsilon' = \frac{1}{\epsilon_{\text{Al}_2\text{O}_3}} + \frac{1}{\epsilon_{\text{Cu}}} - 1 = 0.3972 \quad (5)$$

This accountability has already halved the initial expected wattage, without accounting for in-

insulating layers. Continuing, here's an equation accounting for the decreased wattage per added layer. Note that this is still an approximation using arbitrary parallel planes of identical area, including the planes of the shield layers.

$$P = \frac{\sigma(T_b^4 - T_s^4)}{R_b + R_1 + R_2 + \cdots + R_i + \cdots + R_N + R_s},$$

$R_b =$ Resistance of the emissive body, $\frac{1-\epsilon_b}{A\epsilon_b}$
 $R_i =$ Resistance of the individual shield(s), $\frac{1}{A} + \frac{1-\epsilon_i}{A\epsilon_i}$
 $N =$ The Number of shields being used
 $R_s =$ Resistance of the surface, $\frac{1}{A} + \frac{1-\epsilon_s}{A\epsilon_s}$

(6)

We can simplify this equation significantly by expecting the shields to be made of identical material,

$$P = \frac{\sigma(T_b^4 - T_s^4)}{R_b + N \cdot R_{\text{shield(s)}} R_s},$$

$R_b =$ Resistance of the emissive body, $\frac{1-\epsilon_b}{A\epsilon_b}$
 $R_{\text{shield(s)}} =$ Resistance of the shield(s), $\frac{1}{A} + \frac{1-\epsilon_i}{A\epsilon_i}$
 $N =$ The Number of shields being used
 $R_s =$ Resistance of the surface), $\frac{1}{A} + \frac{1-\epsilon_s}{A\epsilon_s}$

(7)

Substituting in the desired values to (7), and using the simplified assumption that all of the insulating layers will have the same surface area as the alumina block, the respective values are

$$P = \frac{\sigma(T_b^4 - T_s^4)}{R_b + R_1 + \dots + R_N}, \quad (8)$$

$$R_b = \frac{1 - \epsilon_{\text{Al}_2\text{O}_3}}{A\epsilon_{\text{Al}_2\text{O}_3}} = 71.35 \text{ m}^{-2}$$

$$R_i = \frac{1}{A} + \frac{1 - \epsilon_{\text{Ta}}}{A\epsilon_{\text{Ta}}} = 198.6 \text{ m}^{-2}$$

$$N = \text{The number of shields being used}$$

$$R_s = \frac{1}{A} + \frac{1 - \epsilon_{\text{Cu}}}{A\epsilon_{\text{Cu}}} = 7.0 \text{ m}^{-2}$$

Putting these values into table 2 seen below, it's evident that the insulating layers provide dramatic returns on heating efficiency, but that those returns do diminish.

Number of layers	Expected Wattage	Wattage decrease
0	1895	N/a
1	536	-1359
2	312	-224
3	220	-92
4	170	-50
5	138	-31

Table 2: The anticipated wattage for a given number of tantalum layers, and the subsequent reduction in wattage each additional layer facilitates

This approximation has yet to account for the necessary increase in area each enveloping surface must accommodate, since parallel planes may all hold identical areas, but spheres encompassing one another must get progressively larger. This will yield further claimed efficiency gains in our approximation, as is evident from the surface area variable in the resistance terms of (6), (7), and (8). Of additional note is that this approximation would appear to suggest that infinite layers could completely stop the transfer of heat, and violate thermodynamics in doing so, however it's trivial to note that the case of infinite layers would simply be a solid and continuous volume of tantalum, and thus conduct the heat rather effectively in comparison.

3.2 Water Flow Heat Transfer Calculations

The first step in calculating this system's rudimentary cooling needs is to use a steady-flow energy balance equation.

$$\dot{Q}_{in} + \dot{W}_{in} + \sum_{in} \dot{m}\theta = \dot{Q}_{out} + \dot{W}_{out} + \sum_{out} \dot{m}\theta \quad (9)$$

Many of these terms can be eliminated off the cuff. The only energy introduced is the heat, $\dot{Q}_{in}$, of the heating element, and the kinetic energy and work done by the cooling line is negligible, leaving only $\dot{Q}_{out}$. From the sections 3.1 calculations it can be assumed that less than 600 Watts of power will be used to heat the alumina, so this will be used as an overly safe upper bound on the heat which needs to be removed from the system. Lin Hall is equipped with chilled water lines to supply the cooling needs of the building's equipment, so this is our chosen cooling fluid. Because these lines were designed to support multiple applications of similar scale, their limitations will not need to be considered, as later analysis will prove.

Moving forward with these simplifications, we get the following form,

$$\dot{Q}_{in} = c_{p,avg} (T_{outlet} - T_{inlet}) \dot{m} \quad (10)$$

Because the cooling system is oversized, the outlet temperature of the water will be kept to a minimum of 10° C above the inlet, rather than maximizing the cooling potential of the water. Solving this equation provides a $\dot{m}$ value of 14 cm³/s, or 0.84 L/min. To simplify further calculations this will be rounded up to 1 L/min. This volumetric flow rate will be used moving forward.

The next design choice was to pick an appropriate VCR fitting for the given thickness of the copper walls. The corresponding pipe diameter will determine the cross-sectional area of the fluid at any given point, and thus determine the flow velocity. This is an important quantity, as it will later help determine the Reynold's Number, and thus the measure of turbulence within the

pipes. The more turbulent the flow, the better the heat transfer, as more of the fluid is exposed to the outer walls. The Reynold's number also depends on the fluid density, dynamic viscosity, and hydraulic diameter of the line. The hydraulic diameter is the same as the line diameter, so it's also dependent on the choice of VCR fitting. All of these factors were tabulated, then correlated to the pressure drop each line would experience. While it's expected that Lin Hall's chilled water will be sufficient, the variables at play correlate to magnitudes of difference, and higher pressure systems hold higher risks in vacuum. The Haaland equation

$$\frac{1}{\sqrt{f}} = -1.8 \log \left[\left(\frac{\varepsilon/D}{3.7} \right)^{1.11} + \frac{6.9}{Re} \right] \quad (10)$$

was used to approximate the friction factor, f , which was used to calculate the major losses in each possible line through the Darcy Weischbach equation,

$$\frac{\Delta p}{L} = f_D \cdot \frac{\rho}{2} \cdot \frac{\{v\}^2}{D} \quad (11)$$

With all of these in mind, the potential pressure drops (as normalized for a single meter's length) were tabulated, and from these it was determined that 0.18 inches, which reflects the nominal 1/4" VCR standards, was the ideal ID of the line. The model of the heater's walls was then modified to accommodate the lines, with three rows along three of the walls, and two on the wall which contains the VCR connectors. The geometry of this updated model was then used for validation inside the ANSYS Fluent Computational Fluid Dynamics (CFD) modeling process, which is discussed in the next chapter.

4 CFD Analysis and Validation

Using this updated model, the heat flow was simulated using ANSYS Fluent, a computational fluid dynamics (CFD) focused submodule of the finite element analysis (FEA) suite of tools ANSYS offers. The goal of this analysis would be to verify the results of the mathematical analysis, mainly the conclusion that the cooling lines were sufficient for the amount of heat which would ever be applied in anticipated operating conditions. Additionally the work within ANSYS would need to correlate to the values determined by the mathematical analysis. Due to the nature of estimations, these were not expected to be exact matches, just values within a magnitude of the mathematical solution. Additionally, the solution offered by ANSYS was checked for internal consistency, as errors in the setup process could be identified through results which violate simple physical laws, such as matter's inability to be created nor destroyed.

The approximations and simplifications made here do not exactly match those made in the mathematical approach. One constraint limiting the scope of this analysis is that the version of ANSYS freely available to students limits the number of nodes, or elements, used in a given analysis. This limits the fidelity of the results based on the geometry analyzed. As such, superfluous geometry is excised to ensure the majority of elements are 'spent' on the specific volumes which needed to be analyzed. Because this analysis was focused on the efficacy of the water cooling lines, the only model analyzed was the walls which contained said lines. This raises the assumption that the heat held by the shutter and lid is negligible. For the lid this is a simple assumption, as it is held pressed against the walls such that conduction is maintained with the walls, and any heat it captured could easily flow to the walls. The heat retained by the shutter could be considerable, within the scope of this piece of equipment, however the containment of that heat is well accounted for; the wheel-based actuation solution means that very little of the cart could conduct heat to other components, and the radiative source of the heat is either focused upon the substrate or contained in full by an effectively insulating plate. With these considerations taken in mind, the actual properties and techniques of the setup can be considered and discussed.

Because the method of design used CAD files designed inside of Autodesk Inventor, those CAD models were able to be imported directly into ANSYS, albeit converted from their proprietary file format to the more universal STEP format, which is the "Standard for the Exchange of Product model data", a file format for 3D objects defined by the International Organization for Standardization [5]. The file was opened within ANSYS's own geometry application, SpaceClaim, and then processed. SpaceClaim's utility in this step was that it could be used to verify the model's compatibility with ANSYS, label sections of faces and volumes which would be referenced later, and to identify the cavity occupied by the flowing water. This kind of problem is called a conjugate heat transfer problem, a problem dealing with the coupled bodies of a solid and a flowing fluid and their boundary in common [4]. ANSYS Fluent and computational FEA tools more generally are well equipped to solve these kinds of problems, as they are banal expressions of continuum that require rote work to solve.

Having made all requisite preparations, the setup was provided the initial values and constants necessary to effectively solve the problem. Some of these were more simply determined than others. The fluid velocity at the inlet was set to 1 m/s and 300 K, per the water flow heat transfer calculations of section 3.2. At the outlet it was assumed that there was no gauge pressure, so that there was no backpressure or driving pressure considered. These assumptions all relied on the idea presented in section 3.2 that Lin Hall, the building this equipment will reside in, has a cooling water setup which far exceeds the needs of any system which could be designed for these purposes. The acceleration due to gravity was added to the parameters due to its ease of implementation and uncontroversial, if insignificant, effects. Finally, the radiative heat was accounted for. Because of the scope of its functionality, the heat flow boundary conditions for radiation was not used inside of ANSYS. The simplifications made to accommodate this analysis made that an inappropriate choice, and instead heat flux applied across the interior of the walls was used to represent the heat source. The amount of heat flux was determined as,

$$\text{Heat Flux} = \frac{\text{Heat}}{\text{Total Area}} \Rightarrow \frac{\text{Maximum Anticipated Heating Wattage}}{4 \cdot \text{Individual Interior Wall Area}} \quad (12)$$

and using the values below, one determined in section 3.1 and one taken from directly from the model,

$$\text{Maximum Anticipated Heating Wattage} = 600 \text{ W}$$

$$\text{Individual Interior Wall Area} = 8.941 \text{ in}^2 = 0.02307 \text{ m}^2$$

These values provide a heat flux of 26 kW/m^2 . This is an inherently high estimation, as it assumes that at the highest anticipated power delivery the setup is also at steady-state, meaning it has been left on long enough that associated components have reached equilibrium temperatures, and finally it assumes that no heat is lost among the layers of tantalum foil insulation, whose sole purpose is to concentrate heat at the substrate and thus away from reaching the cooling walls.

Having assembled all prerequisites, including those correlated through or provided by prior work in this process, the most pressing questions can be answered. Firstly, what temperatures does the block reach?

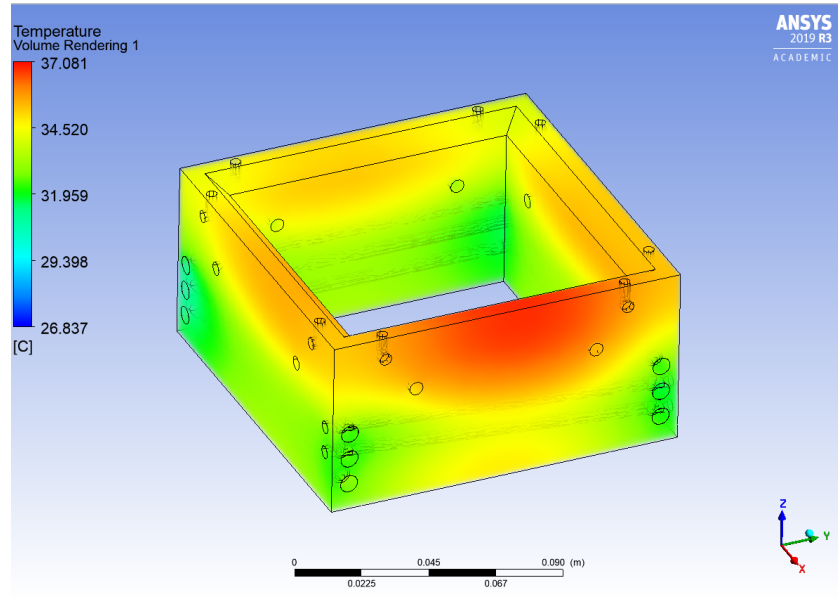


Figure 7: A volume rendering of the walls' temperature at a steady-state operating condition defined in the text.

Figure 7 shows the approximated distribution of temperature at steady-state. From the legend's upper bound one can see that the highest temperature seen within the copper block is 37°C . The lower bound is not informative, as it's simply the temperature of the chilled water supply, which is among the predetermined boundary values. From this representation it's clear that the temperature should never get so great that the water lines struggle in the slightest. It's also clear from the model's heat distribution that the wall with two lines, rather than the three contained by the other walls, will be the hottest because of its lesser capacity for cooling. This is consistent with anticipated behavior, and thus increases confidence in the accuracy of this model. In addition, the walls are coolest where they're in contact with the cooling lines. The validity of this model, and its parameters, will be further reinforced later in this section.

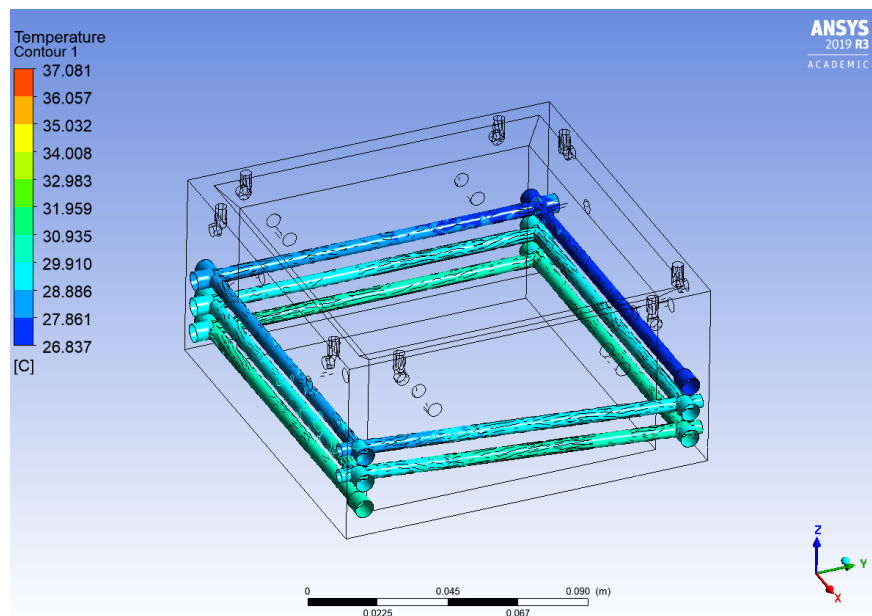


Figure 8: A rendering of the surface temperature within the cooling lines during operating conditions.

Figure 8 is also consistent with the behavior expected for the system. At the inlet it's at its coolest, and as the water follows the perimeter of the walls it gradually heats up.

Being satisfied with the validity of the results so far, the final result to explore is the pressure at the outlet. The water flow calculations emphasized a turbulent flow, to maximize the efficacy of the water's heat transfer. This meant that the driving pressure of the system needed to be just

enough to result in positive flow. In practice this may not make much of a difference, but for the purposes of exploring the theoretical capabilities of a system like this it's a more than useful exercise. Looking at the average pressure across the outlet and referencing ANSYS's legend reveals a negative pressure. Noting that this is a relative pressure, one can draw the conclusion that the water flow values have just overshoot the initially calculated expectations. But, also noting the magnitude of the outlet pressure against the magnitude of the driving pressure, and accounting for the additionally imbued work of gravity and the height differential, it's reasonable to assume this correlation between the mathematical approximation and the CFD approximation is indicative of a valid model.

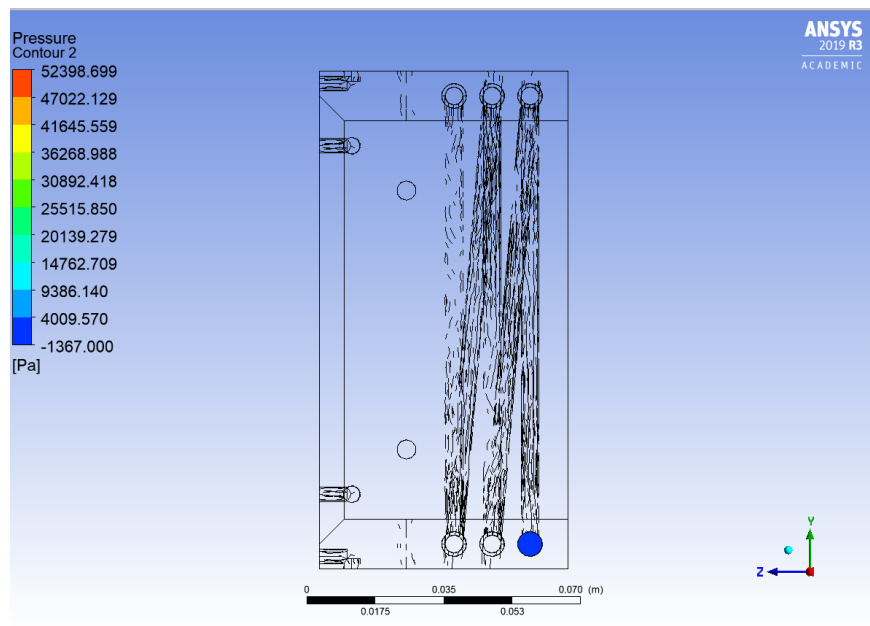


Figure 9: A rendering showing the simulated water pressure at the outlet while operating, notably approximating the average pressure across the entire outlet rather than delineating areas of high and low pressure.

5 Use, Troubleshooting, and Future Application

5.1 Intended Use Procedure

The heater's design, and the provided procedure, is focused on use within our existing setup. However, the majority of procedures should be universal, or at least easily adapted, as vacuum chamber setups for PVD broadly operate on alike principals.

WHILE OPEN TO THE ATMOSPHERE

To access the substrate's mounting point, all a user will need to do is remove the lid from the assembly. This is more complicated than most lids, but still eminently doable. First, one needs to ensure the heat sink hardware has been properly secured. Briefly disconnecting it from its actuating assembly, where it's connected to a feedthrough, should be enough to do this. Next, one will need to free the lid by setting aside the lid tightening hardware. This can be accomplished by first removing the eight screws securing the four pieces of lid tightening hardware, and then the four pieces of hardware itself. A screwdriver can then be used to pry the lid off of the walls which it's resting on, using the slot designated for prying. Every effort should be made to lift the lid vertically, to keep the lid's stacked layers of tantalum foil from hitting those of the walls. Once the lid is clear from the rest of the assembly it can be inverted, revealing the alumina block where the substrate can be clipped into place. Following these steps in reverse order, one can reinstall the appropriate components and then prepare for the sealed procedures.

WHILE SEALED

Before the roughing pump is enabled, the user will need to ensure all components are in their appropriate places. The following checklist should generally be observed:

- The shutter-cart needs to be placed directly beneath the assembly.
- The heat-sink-block needs to be displaced so that it's not removing heat during the warming-up process.
- The cooling lines should have water flowing through them.

5.2 Troubleshooting

These steps ensure not only that the alumina and substrate are efficiently heated, but that radiant heat isn't inadvertently released into the chamber, possibly damaging it.

Having verified these steps, the heater can be safely initialized during the roughing process. It's important to begin heating while roughing, as the heat will cause various pieces of equipment to off-gas, and these gases are best handled by the roughing pump. The goal of this pump down procedure is to absolutely minimize the gas captured by the cryo-pump. Cryo-pumps condense gases using extremely cool liquids (such as liquid helium or nitrogen) and "trap" the gases (now deposited as solids) for the duration of operation. However, the cryo-pump can become saturated, or "full", and cease removing gases from a vessel, once its cooled surface is obscured by enough deposits. Roughing pumps generally remove gas to the surroundings, and don't face this limitation. They're instead limited by the pressure they can exert on the chamber, never being able to reach the higher levels of vacuum required for sufficiently advanced deposition. As such, it's imperative to begin heating during the roughing process, so that the majority of off-gases can be removed by the roughing pump.

Once the heater has reached a steady-state at its desired temperature, and the deposition sources are evaporating at a sufficient rate, the shutter-cart can be moved out from under the assembly and the thin film deposition can begin. Once the film has

5.2 Troubleshooting

Because this design only exists on paper, it is impossible to now know all issues it may encounter in installation. However, the potential for some issues has been anticipated, with proposed solutions for those issues as well. Consideration needs to be given to the fact that this design focuses purely on the physical components, and does not delve into the specifics of the wiring needs, or other less tangible choices. This has been done in part for the sake of simplicity, but more broadly in light of the patent needs and implementation of such subsystems.

Of note is the potential for off-gassing in the form of particulates released by the material when two parts slide against one another. Like a microscale version of any machining operation, this

removed material enters the chamber and effectively acts as a gas, even if in reality it's merely an aerosolized solid. Almost all of its negative effects, at the very least, are those same negative effects which are the very reason the chamber is brought to a vacuum in the first place. The use of lubrication, which would provide a film of fluid to interface between any sliding bodies, is not possible here, as what constitute lubricants available at this scale of manufacture are all given to off-gassing, the very problem that needs mitigation. This problem guided many of the decisions made in this process, but certain compromises were made too. The cable which controls the position of the shutter-cart could incite this issue, as the static friction between it and the various pulleys it interfaces with may slip into kinetic friction and cause this issue. That is why the cables need to be held taut, to reduce the possibility of this occurrence. Additionally, should tightening these cables fail to solve the issue, there is an additional provided design for an actuation solution with phalanges-like rods rotating through bearings as revolute joints. Additional sources of frictional removed material might be the tight fit between the top of the shutter-cart and the bottom of the top heater. Possible interference here would be solved through additional machining to reach the desired fit, or preferably adjusting the vertical position of the shutter-cart and rails through their mounting points on the heater support.

Additionally, and far more obviously, nixing the custom actuary components in favor of commercial options, and swapping in a linear feedthrough is a simple solution, albeit one with a price-tag several hundred-times the materials budget for the designs provided herein, which rely on currently owned rotary feedthroughs.

Should the cart prove difficult to actuate with the initial cable setup, there are provided alternative pulley sizes, to accommodate reducing, or increasing, the number of rotations required to move the shutter-cart. If this fails to solve the problem, once again, there is the potential to instead use a linear feedthrough to actuate the cart.

5.3 Future Applications

This setup will be used for thin-film depositions for the foreseeable future, and some of the accommodations this could require have already been accounted for. For example, the significant heating requirements of a process like gold deposition, and deposition onto gold, were used as an upper bound during the design process. Beyond this, the assembly is compartmentalized such that many of the components can be replaced with correlating alternative parts.

A process not covered by the broader parts of this document was the installation of an additional 3 ConFlat® 2-3/4 flanges around the 'neck' of the base well, which allow for another 3 feedthroughs but more importantly 3 feedthroughs which are well positioned for the installation of an electron gun, and thus raising the possibility of e-beam evaporation within the chamber. This additional method would be compatible with the heater.

6 Conclusion

Solving, or attempting to solve, the numerous problems presented by the needs of thin film manufacture presents a daunting challenge. The design provided with and elaborated upon in this document hopefully solves many of these problems, or at least provides an appropriate starting point for solving those problems, and a direction to take additional steps in the future. Unfortunately this was not able to be built and tested empirically within the scope of this project, due to constraints in both time and available labor, as the covid-19 pandemic slowed much of daily life.

7 Acknowledgments

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8 Drawings

9 Appendices

References

- [1] 1960 - *Metal Oxide Semiconductor (MOS) Transistor Demonstrated*. URL: <https://www.computerhistory.org/siliconengine/metal-oxide-semiconductor-mos-transistor-demonstrated/> (visited on 06/20/2020).
- [2] Y. Kuo. “Thin Film Transistor Technology—Past, Present, and Future”. In: *Interface magazine* 22.1 (Jan. 2013), pp. 55–61. DOI: 10.1149/2.f06131if. URL: <https://doi.org/10.1149/2.f06131if>.
- [3] *Kurt J. Lesker Company*. [Online; accessed 27. Jun. 2020]. June 2020. URL: https://www.lesker.com/newweb/deposition_materials/materialdepositionchart.cfm?pgid=0.
- [4] T. L. Perelman. “On conjugated problems of heat transfer”. In: *Int. J. Heat Mass Transfer* 3.4 (Dec. 1961), pp. 293–303. ISSN: 0017-9310. DOI: 10.1016/0017-9310(61)90044-8.
- [5] Michael J. Pratt. “Introduction to ISO 10303—the STEP Standard for Product Data Exchange”. In: *J. Comput. Inf. Sci. Eng.* 1.1 (Mar. 2001), pp. 102–103. ISSN: 1530-9827. DOI: 10.1115/1.1354995.
- [6] Donald L. Smith. *Thin-Film Deposition Principles & Practice*. McGraw-Hill, Inc., 1995. ISBN: 0-07-058502-4.
- [7] Robert K. Waits. “Edison’s vacuum coating patents”. In: *Journal of Vacuum Science & Technology A* 19.4 (2001), pp. 1666–1673. DOI: 10.1116/1.1326948. eprint: <https://doi.org/10.1116/1.1326948>. URL: <https://doi.org/10.1116/1.1326948>.