

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

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

A THESIS APPROVED FOR THE
SCHOOL OF AEROSPACE AND MECHANICAL ENGINEERING

BY THE COMMITTEE CONSISTING OF

Dr. Yingtao Liu

Dr. Lloyd Bumm

Dr. Hamidreza Shabgard

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Abstract

1 Introduction

The Necessity of High Vacuum/UHV for thin film (and other) applications, where heating structures fit into the picture

The motivation to custom-build a solution and the nature of vacuum chambers for scientific applications (that is, some of them are surprisingly old, such as our chamber for example)

Vacuum chambers, and the successful maintenance of vacuum, have played a fundamental role in the technological revolution of the last one-hundred years. Without vacuum, we would not have the light of filament bulbs, cups capable of keeping coffee hot for hours, or any of the billions of transistors which now freely occupy nearly every device around us. The technology to produce vacuum has been a necessary stepping-stone in nearly every major scientific endeavor which has rendered our modern world. Vacuum is so ubiquitous that

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}	$> P$	

As we know, "Nature abhors a vacuum," so good equipment and techniques are needed to create one [1]

2 Design Outline

Figures from CAD renders throughout

The 'base' of this system was a Varian 3118 vacuum evaporator, produced in XXXX for use at XXX Bell laboratories. Because of the fundamental simplicity of a vacuum chamber, and their unwavering expense of manufacture, this system has been used for many decades under different research bodies. But because of this continuous use, this system has been frequently modified during its service, making it far different than the stock system. What remains of the system are its frame, base well, bell jar, and pneumatic lift (to raise and lower the bell jar). Assorted accessories also accompanied the setup, but these are less relevant to my application.

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 XXXcitationsOnDepositionValues. At this temperature range it is very easy to incidentally emit enough heat to damage or even crack the enclosing bell jar. Keeping in mind that even a low vacuum environment severely reduces convective heat transfer, and it's easy to see how thermal runaway poses a serious risk to the operative capacity of the setup.

2.1 per-component discussion and highlights

2.1.1 Water Cooled Walls

Almost all of the system's components are encompassed by the thick copper walls which makeup the outside of the 'box'. Because of the potential hazards posed by the system's radiated heat, the high thermal conductance of the copper makes it ideal as a sink material. Additionally, copper's machinability mean that more complicated manufacturing operations can be employed, and special capabilities can be leveraged.

Water cooling is commonly employed in vacuum evaporators, but these systems rely on tacked on pipes, or made-to-order cooled walls where the desired cooling line is milled into steel plate before being rolled into the desired chamber curvature. These solutions are beyond costly, and would require the replacement of one of the system's most valuable inherited components, the physical chamber itself. Instead, this system uses intersecting through holes to establish a 'pipe' for cooling water, and welded corks to seal the end of each hole. In this way the benefits of the embedded cooling lines are gained, but without the significant manufacturing cost.

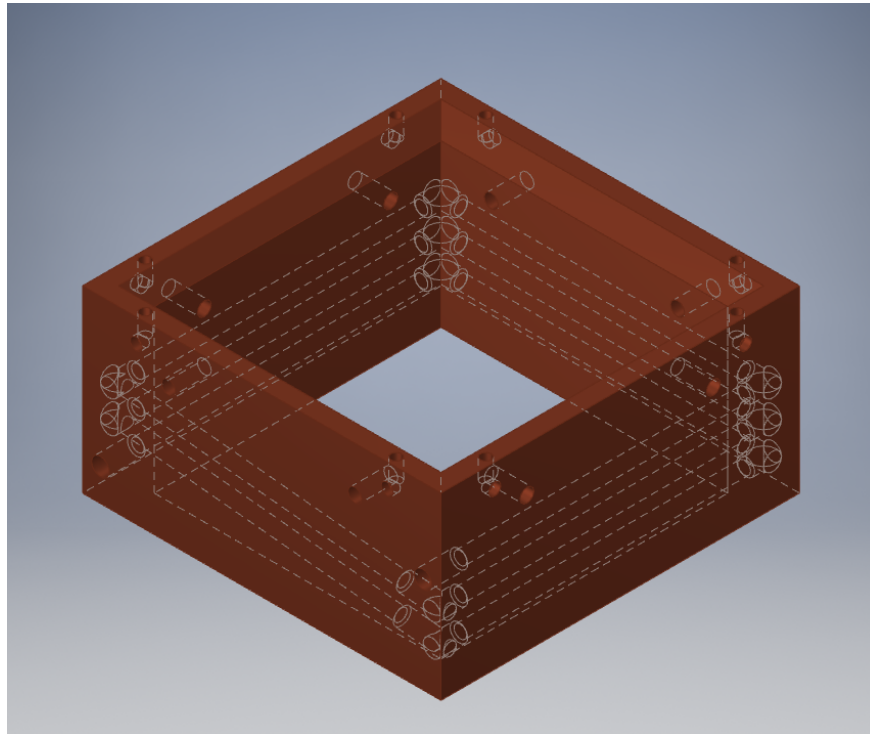


Figure 1:

A discussion of the CFD modeling techniques used to validate this design is found in section 2.2.2.

2.1.2 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's sized to ease prying the lid off the box's top.

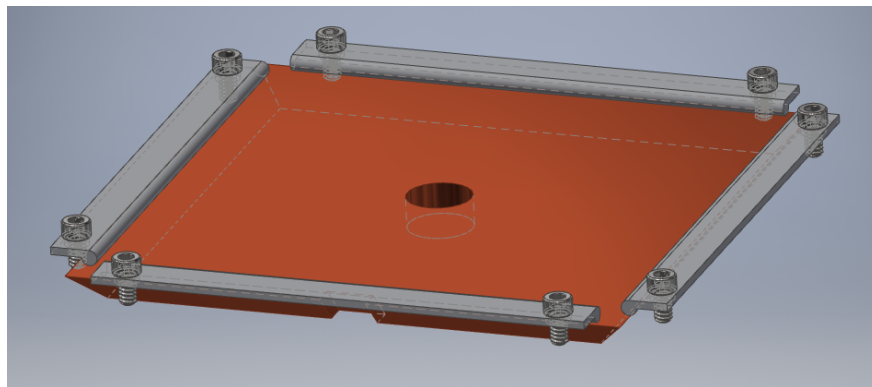


Figure 2:

2.1.3 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 maybe 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. Much like an active high switch, when the spring isn't displaced it presses the sink against the

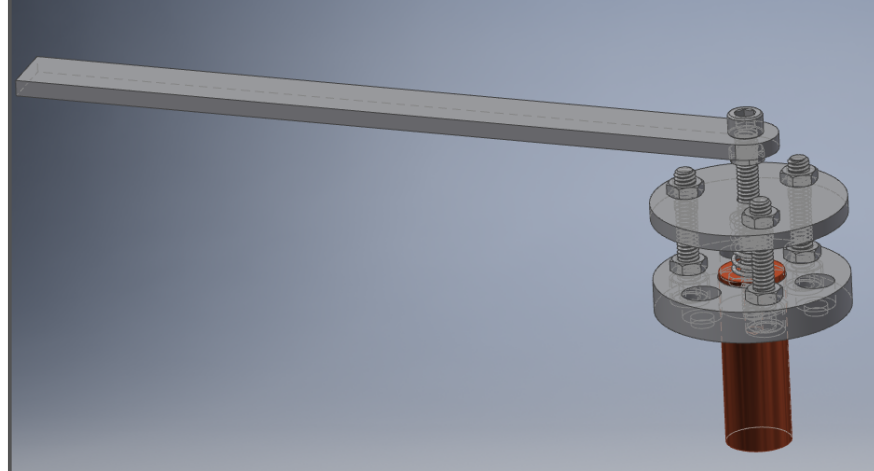


Figure 3:

2.1.4 bottom shutter

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.

The processes of heating and cooling the substrate take up large amounts of time in a given deposition trial. This shutter seeks to reduce the time it takes to heat up the substrate. 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.

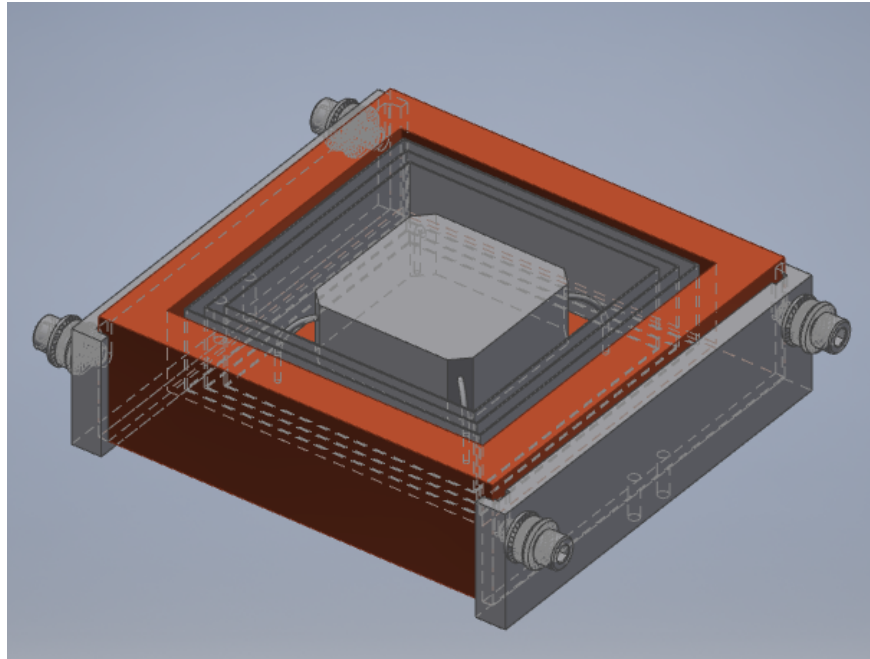


Figure 4:

2.1.5 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 less than XXX, 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.

In 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.

2.1.6 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 XXX.

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

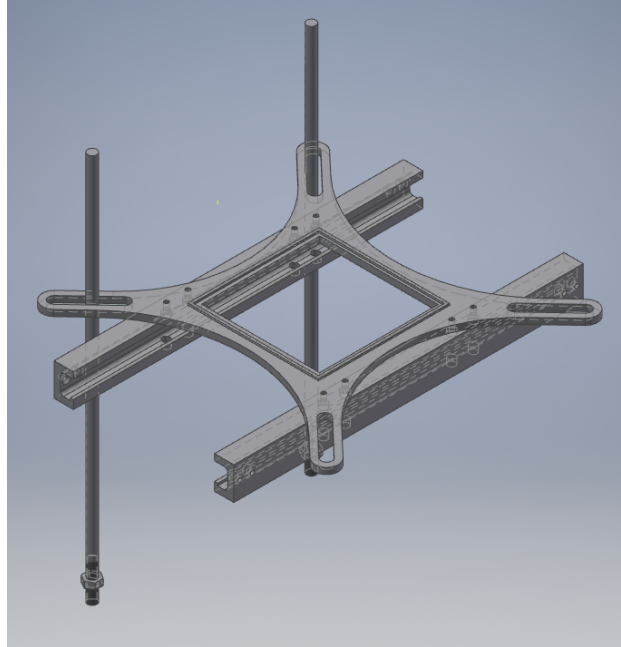


Figure 5:

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

2.1.7 etc.

2.2 Mathematical basis for choices

2.2.1 radiative heat transfer

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 in^2 (0.0193548 m^2), 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

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

$P \equiv$ Power Radiated, W
 $A \equiv$ Surface Area, m²
 $T \equiv$ Absolute Temperature, K

This initial estimate offers

$$P = 2883.49\text{W}$$

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 my personal 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. We'll start with 25° C, standard temperature.

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

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

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

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. We will account for this discrepancy later

The next assumption is that we'll use layers of tantalum foil to reduce the radiative losses of our block. In conduction analysis we use thermal 'resistance', in analogy to electrical resistance, to calculate the reduction in heat transfer in a system. While we're far, in this approximation, from accounting for conductive heat transfer, we can still use this technique for 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, highlighting the shortcomings of this approximation method.

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

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 insulation's efficacy is reduced.

Emissivities are a fundamental component of our approximation. These coefficients, ϵ , work like the view factor, except they're independent of our final geometry. They're merely functions of what materials each body is made of, and sometimes what temperature as well. We'll start with the high-end approximate values for these, so that our approximation favors more power required, not less.

Pulling from the table provided here [http://www-eng.lbl.gov/dw/projects/DW4229_LHC_detect_or_analysis/calculations/emissivity2.pdf] we have a number of applicable emissivities.

$$\begin{aligned} \epsilon_{\text{Al}_2\text{O}_3} &= 0.42 \\ \epsilon_{\text{Ta}} &= 0.26 \\ \epsilon_{\text{Cu}} &= 0.88 \end{aligned}$$

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 our initial expected wattage, without accounting for insulating layers. Continuing, here's a table 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.

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

$$\begin{aligned} R_b &= \text{Resistance of the emissive body, } \frac{1-\epsilon_b}{A\epsilon_b} \\ R_i &= \text{Resistance of the individual shield(s), } \frac{1}{A} + \frac{1-\epsilon_i}{A\epsilon_i} \\ N &= \text{The Number of shields being used} \\ R_s &= \text{Resistance of the surface), } \frac{1}{A} + \frac{1-\epsilon_s}{A\epsilon_s} \end{aligned} \quad (6)$$

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

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

$$\begin{aligned} R_b &= \text{Resistance of the emissive body, } \frac{1-\epsilon_b}{A\epsilon_b} \\ R_{\text{shield(s)}} &= \text{Resistance of the shield(s), } \frac{1}{A} + \frac{1-\epsilon_i}{A\epsilon_i} \\ N &= \text{The Number of shields being used} \\ R_s &= \text{Resistance of the surface), } \frac{1}{A} + \frac{1-\epsilon_s}{A\epsilon_s} \end{aligned} \quad (7)$$

Substituting in our desired values to (7), we get.

$$\begin{aligned}
 R_b &= \frac{1-\epsilon_{\text{Al}_2\text{O}_3}}{A\epsilon_{\text{Al}_2\text{O}_3}} = 71.35 \\
 P &= \frac{\sigma(T_b^4 - T_s^4)}{R_b + R_1 + \dots + R_N}, \\
 R_i &= \frac{1}{A} + \frac{1-\epsilon_{\text{Ta}}}{A\epsilon_{\text{Ta}}} = 198.6 \\
 N &= \text{The Number of shields being used} \\
 R_s &= \frac{1}{A} + \frac{1-\epsilon_{\text{Cu}}}{A\epsilon_{\text{Cu}}} = 7.0
 \end{aligned} \tag{8}$$

Putting these values into a table, seen below, it's evident that the insulating layers provide dramatic returns on heating efficiency.

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

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).

$$\begin{aligned}
 P &= \frac{\sigma(T_b^4 - T_s^4)}{R_b + R_1 + R_2 + \dots + R_i + \dots + R_N + R_s}, \\
 R_b &= \text{Resistance of the emissive body, } \frac{1-\epsilon_b}{A_b\epsilon_b} \\
 R_i &= \text{Resistance of the individual shield(s), } \frac{1}{A_i} + \frac{1-\epsilon_i}{A_i\epsilon_i} \\
 N &= \text{The Number of shields being used} \\
 R_s &= \text{Resistance of the surface, } \frac{1}{A_s} + \frac{1-\epsilon_s}{A_s\epsilon_s}
 \end{aligned} \tag{9}$$

2.2.2 water flow and conductive heat transfer

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

$$\dot{Q}_{in} + \dot{W}_{in} + \sum_{in} \dot{m}\theta = \dot{Q}_{out} + \dot{W}_{out} + \sum_{out} \dot{m}\theta$$

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 radiativeheattransferXXX calculation 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,

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

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 $14 \frac{\text{cm}^3}{\text{s}}$, or $0.84 \frac{\text{L}}{\text{min}}$. To simplify further calculations this will be rounded up to $1 \frac{\text{L}}{\text{min}}$. 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

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

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,

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

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 0.25 inch VCR standards, was the ideal OD 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. Using this updated model, the flow was simulated using ANSYS Fluent

This analysis substantiated the earlier estimations, confirming that at steady state the pipe flow would keep the temperature of the outer walls below 40° C throughout

XXX

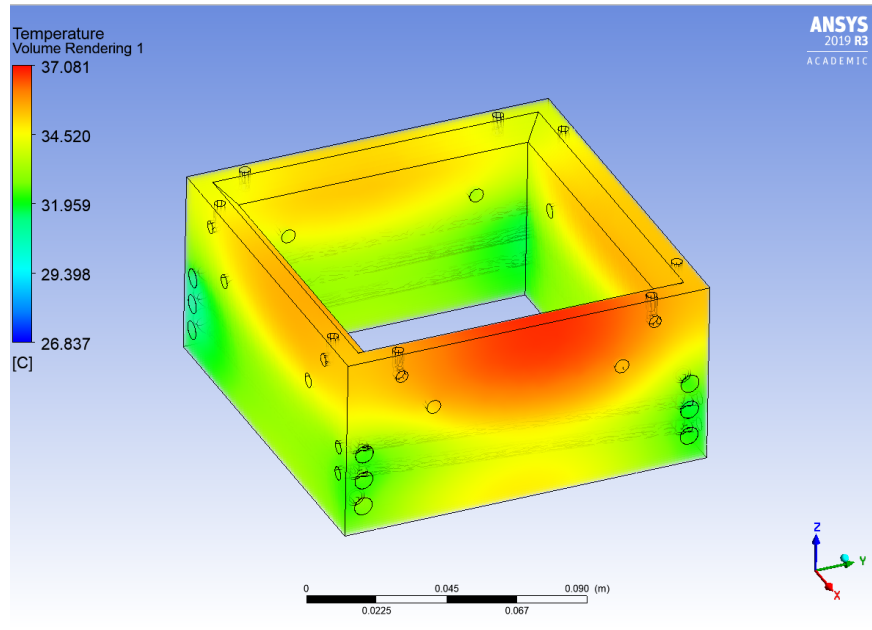


Figure 6:

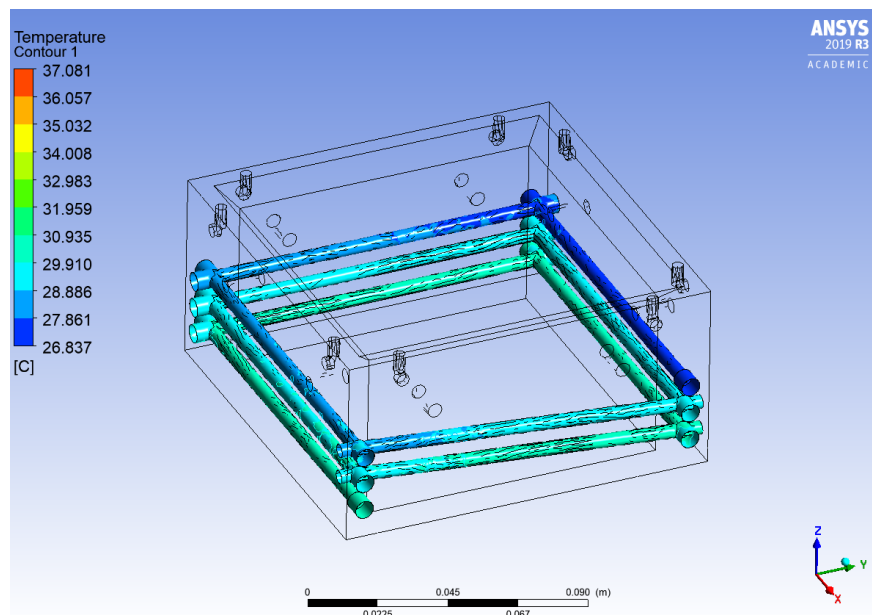


Figure 7:

2.3 Machining and manufacturing basis for choices

Why certain designs are easier to machine than others, and how my choices reflect simpler manufacturing techniques

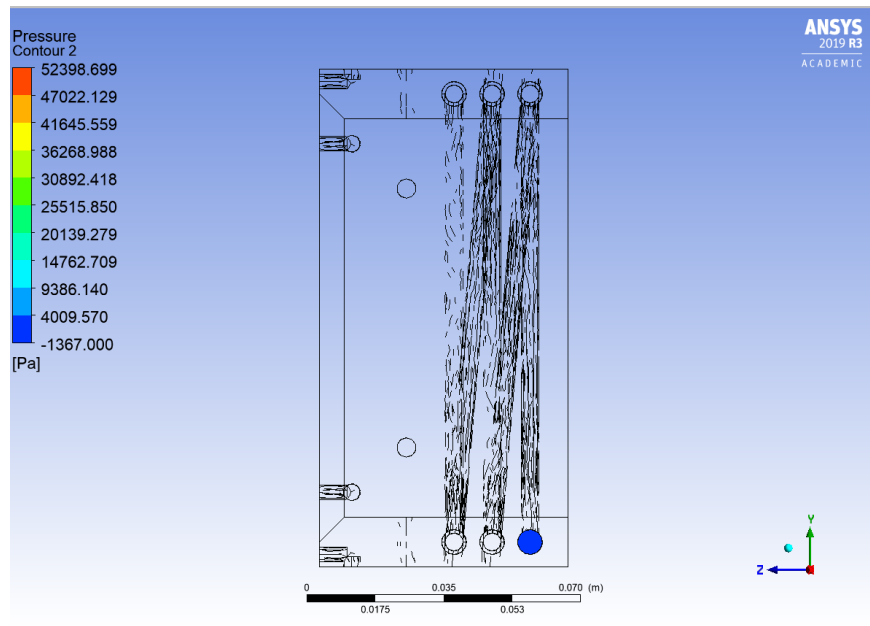


Figure 8:

2.3.1 Discussion of vacuum part standards

Talking about Conflats, VCR, outgassing ratings, etc. here

The ubiquity of high and ultrahigh vacuums in physics experiments means that there is a demand for vacuum compliant equipment, and where there is a demand there is an industry built to meet it. And where there is an industry built to service a technical need, there are standards built to accommodate interoperability of components. This setup takes advantage of a number of these standards, most notably ConFlat® and Swagelok VCR® fittings. ConFlat fittings offer

2.3.2 sourcing high vacuum/UHV compliant components

Talking about vacuum distributors and used/refurbished market here

Multiple online retailers exist to furnish vacuum-appropriate components. Alternatively, the long history of vacuum use in technical applications means there is a fully saturated second-hand market, including dedicated resellers and listings on auction sites, such as eBay. These can offer significant savings when compared to brand-new equipment, but often come with certain caveats. Some pieces of vacuum setups are custom-made to accommodate the intended use, and these customizations carry over moving forward, potentially with unforeseen consequences.

3 Application and use-case, outline of operating procedure

Use-case, examples of applications for thin films made in-house

- get some examples of these

Checklist prior to operation

1. *comfortable amount of pressure applied to heater I guess*

Rough Operating procedure:

1. *Once roughing procedure has begun, begin running current through the heating element. This will allow off gassing before the cryo-pump, which can only collect a finite quantity of gasses, so it's imperative to maximize the effectiveness of the roughing pump.*
2. *Ensure the heatsink block has been displaced from the Alumina, ie that the feedthrough which manipulates it has been extended and locked from the outside.*
3. *Once block has reached appropriate temperature, as measured by embedded thermistor,*

test

test

4 Discussion/troubleshooting of potential issues

5 Discussion of future proofing

How my design accommodates the potential for change. Highlighting the potential introduction of ion beam sputtering (additional flanges in base well), modularity and flexibility of components

6 Conclusion

7 Drawings

8 Appendices

References

- [1] Donald L. Smith. *Thin-Film Deposition Principles & Practice*. McGraw-Hill, Inc., 1995. ISBN: 0-07-058502-4.