Lab 2 - Vacuum Assembly and Troubleshooting

Pre-Lab Reading:

Nanotechnology operates at the atomic and molecular level, demanding an environment with minimal disruption. Here, **vacuum** systems become crucial. These systems function by evacuating a chamber, significantly reducing the number of molecules present. This creates a vacuum, a critical element for various nanotechnology processes.

Minimizing **contamination** is paramount. Even a single foreign molecule can disrupt the delicate assembly of nanostructures or skew the results of material analysis. Vacuum systems significantly reduce air molecules, which dramatically reduces the risk of contamination from this source. This ensures the integrity of the nanomaterials being studied or created.

Beyond the basic absence of air, the concept of **mean free path** is important to the integrity of nanofabrication techniques. This refers to the average distance a molecule travels before colliding with another molecule. In a high-pressure environment like air, the mean free path is miniscule, and ironically at the nanoscale, leading to frequent collisions that hinder precise manipulation. A vacuum, however, significantly extends this mean free path. With fewer collisions, researchers can precisely control the movement of atoms and molecules during processes like **thin-film deposition**. In thin-film deposition, materials are meticulously deposited atom by atom. This level of control allows for the creation of uniform, high-quality nanostructures with specifically desired properties.

Safety is another crucial aspect to consider. While vacuum systems themselves aren't inherently dangerous, specific pump types require safety considerations. **Diffusion pumps**, for example, often utilize mercury vapor to achieve ultra-high vacuum levels. Mercury is a hazardous material, and proper handling and disposal procedures are essential to avoid health risks. Similarly, **turbo pumps** operate at high speeds, exceeding tens of thousands of revolutions per minute. These high speeds necessitate proper maintenance to prevent accidents.

Beyond these specific examples, proper training and safety protocols are paramount when working with vacuum systems. This includes understanding the potential hazards of the pumps and materials used, along with safe operation procedures. These measures are vital to protect researchers and maintain a safe working environment.

In essence, vacuum systems are the foundation for a clean, controlled, and low-pressure environment that underpins the world of nanotechnology. From minimizing contamination and enabling precise control through the concept of mean free path, to navigating safety considerations, every aspect of a vacuum system plays a vital role in unlocking the potential of this revolutionary field.

Objective:

The objective of this lab is for students to learn the applications of vacuum technology, and practice assembling and troubleshooting the Kurt J. Lesker Low-Cost Vacuum Trainer. Students will be practicing on a system designed to mimic those found in various industries that utilize vacuum technology, in addition to systems designed by Kurt. J. Lesker. The educator will provide a detailed experimental appendix for students to follow to perform the process.

Background:

In nanotechnology, vacuum is one of the most critical concepts incorporated into various processes and systems throughout the field. Without vacuum, many of the products that we have today simply would not exist, from fishing lures to smartphones. As stated in the pre-lab reading, vacuum allows for direct control over the atmosphere in which these processes are done, permitting manufacturers to regulate conditions within a process chamber, from reducing the overall pressure to dictating the exact gas composition. However, before venturing further into the methods in which vacuums are used, it is important to understand the ways that vacuums are created and maintained.

There are various methods for generating a vacuum, each with its own benefits and drawbacks. Vacuums are created using **vacuum pumps**, which are essentially one-way valves for gas molecules. It is important to understand the different pumping methods and how they are used so that the proper choice can be made when designing and building a vacuum system.

The most basic of these methods is the oil-sealed **rotary vane pump**, as seen in Figure 2.1. These are some of the cheapest and most robust vacuum pumps that can be purchased, for around $1,000-3,000. Depending on the cost and quality of the pump, these can generate a vacuum of around 10^{-2} to 10^{-3} Torr, which is what is referred to as a “Rough” vacuum. While these may be a solid choice for a rough vacuum system, these pumps are not without their problems. One of these issues is known as back streaming, which is where the oil used to create the air-tight seal in the vacuum pump begins to vaporize, finding its way into the process chamber. This has the potential to disrupt and destroy any processes occurring within the vacuum chamber, making these pumps a second choice for many processes that require clean chambers. When an oil-based pump, or **wet pump**, cannot be used, typically a **dry pump** is used instead. Dry pumps do not use any oil to create a seal and instead use gaskets and very tight fittings to generate a seal to create vacuum. An example of one of these dry pumps is known as a **scroll pump**, which uses two spirals, one inside the other, to create a vacuum. While avoiding the issue of back streaming that is found in wet pumps, dry pumps are much more expensive; typically costing more than $10,000.

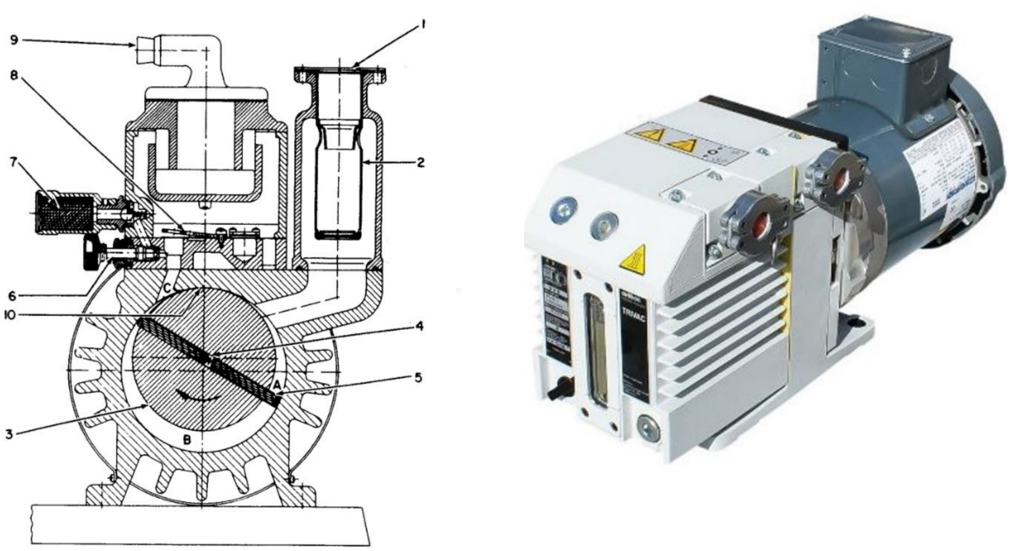


Figure 2.1: Cross-sectional view (left) of a rotary vane pump (right)

Once a **rough vacuum** is established, it is then possible to begin creating a **high vacuum** system. The pressure range for a high vacuum system is usually considered to be around 10^{-2} to 10^{-7} Torr, which can only be generated via the use of specialized pumps. Two of these pumps are turbomolecular pumps, and diffusion pumps, seen in Figure 2.2. These two pumps are nearly identical in operation to jet engines, minus some small adjustments. A turbomolecular pump is effectively a jet engine without the fuel. These pumps work by spinning a set of blades at a rate of anywhere between 20,000-50,000 RPM, depending on the size of the pump. These pumps are very effective at generating a high vacuum, however they come with the risk of being ‘dumped.’ Therefore, it is important to know the pressures that each pump can work at, as outlined in Table 2.1. This means that if these pumps are used improperly, they can destroy themselves from the inside out, causing them to need to be replaced in their entirety. On the other hand, a diffusion pump cannot be ‘dumped’ as it has no moving parts and is overall incredibly simple in its design. A diffusion pump utilizes burning oil to generate the vacuum, similar to how a jet engine would work without its blades. However, since there is oil involved in the operation of the pump, there is a significant risk of back streaming, causing this type of pump to be mostly phased out by turbo pumps.

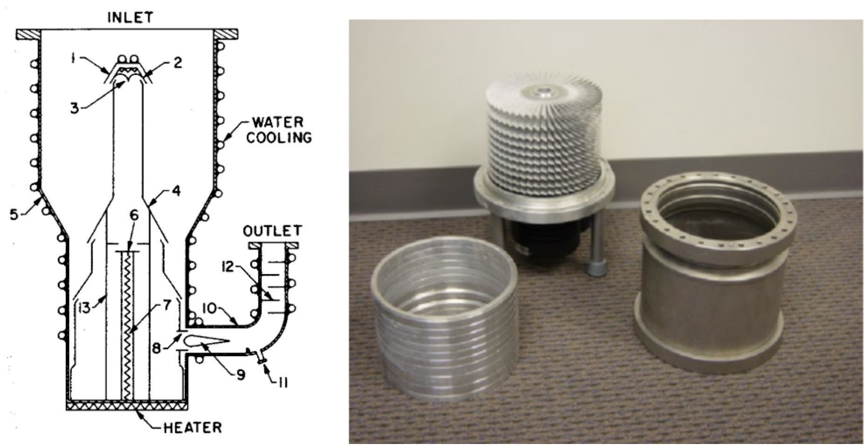
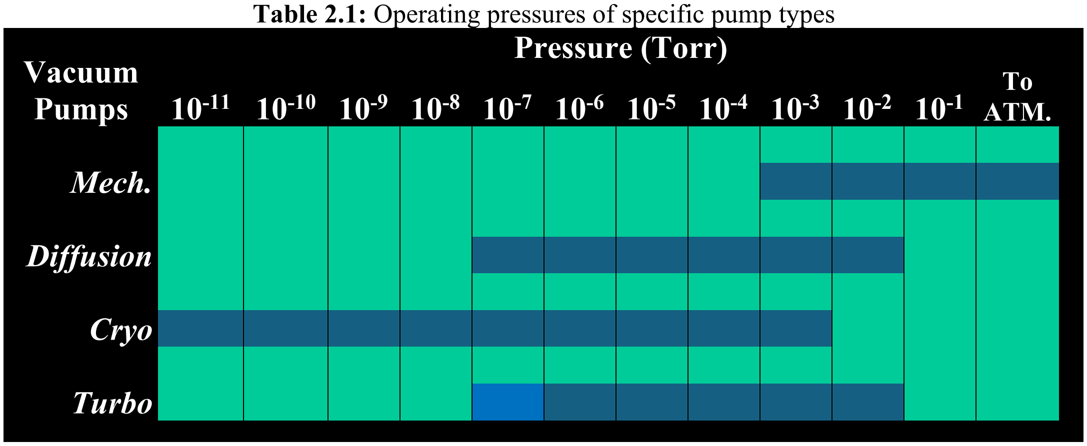


Figure 2.2: Cross-sectional view of a diffusion pump (left). Disassembled turbomolecular pump (right)

Table 2.1: Operating pressures of specific pump types



Once a vacuum system has been developed, it will likely have errors that will lead to leaks or other issues within the system. There can be any number of these problems, as outlined in Figure 2.3. However, it is important to distinguish these problems from each other and understand the underlying reasons they occur.

One of the most evident and logical problems behind vacuum systems are leaks. These can occur in two forms, real and virtual. **Real leaks** are quite trivial to understand, as they are simply the presence of a defect that results in the outside atmosphere finding its way into the vacuum chamber. These can be caused by various reasons, such as incorrectly placed fittings, bad O-rings, or cracks in the piping itself. Since these are relatively easy to understand leaks, it stands that these are also easy to fix, such as realigning the fittings, replacing O-rings, or sealing the hole in the piping. On the other side, **virtual leaks** are also present in vacuum systems. These leaks are not ‘real’, as there is no outside atmosphere entering the vacuum chamber. Instead, these leaks originate from inside of the chamber itself. These can result from air pockets formed inside of screws, to water evaporating and becoming vapor. These leaks do not have an impact on the ending pressure of the vacuum chamber, but they do impact the time it takes for the system to reach that pressure. Therefore, it is important that a vacuum chamber is clean and dry, to minimize the amount of potential virtual leaks.

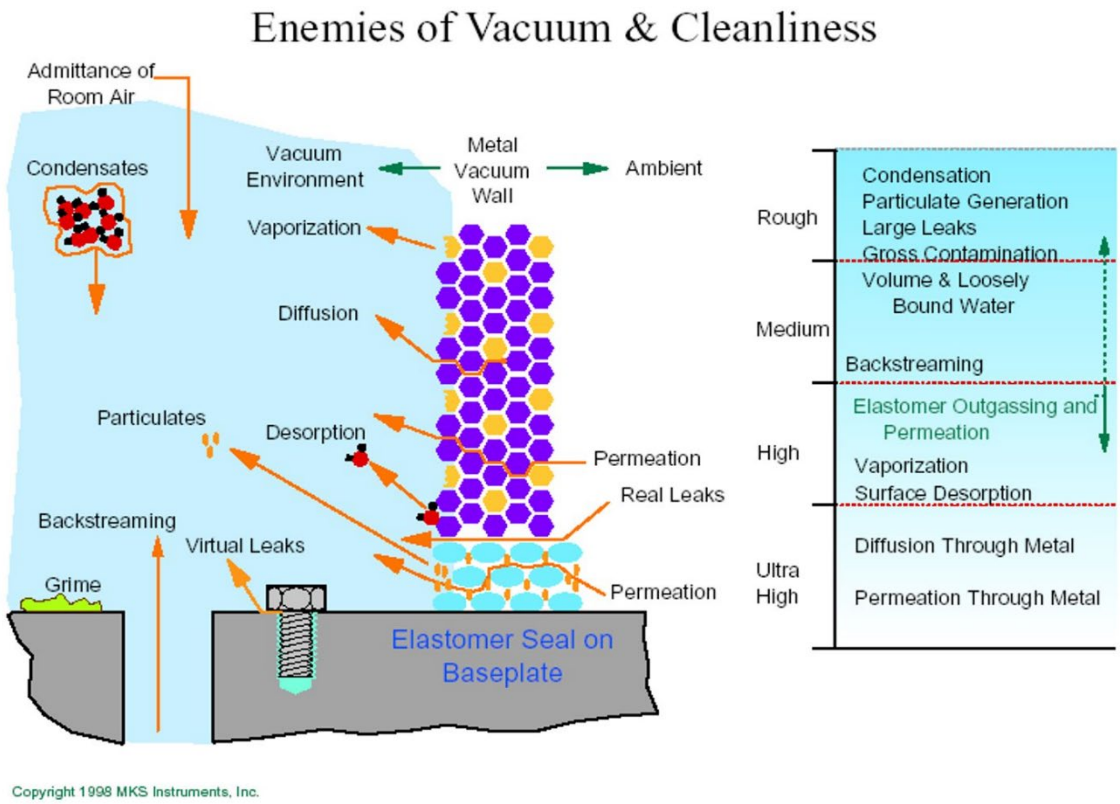


Figure 2.3: Graphic showing various problems with vacuums

Now that the underlying causes of both real and virtual leaks have been outlined, it is time to understand the methodology behind diagnosing these leaks and locating them. In industry, it is common to find dedicated tools for pinpointing the location of leaks using **helium leak detection**. These systems connect to a tool through a designated valve, and once secure, a technician utilizes a helium nozzle to spray helium around locations where a leak is likely to occur. The helium then works its way back to the leak-detection system, which alerts that helium has been detected in the system. However, these tools can be expensive to use, so cheaper methods can be employed in specific scenarios. For example, isolating various sections of the vacuum system from the pump and observing any possible rises in pressure can help to locate potential leak areas. Another economic method of locating leaks is using a can of compressed air to spray around fittings, then waiting to observe any increases in pressure on the gauges. In some cases where the vacuum level can be graphed, one can easily identify a real or virtual leak simply by observing the shape of the graph, as can be seen in Figure 2.4.

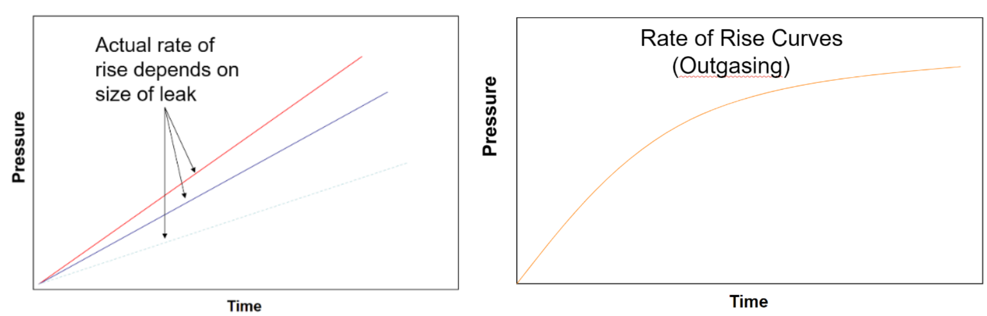


Figure 2.4: Graphs of real (left) and virtual (right) leaks

Experiment:

SAFETY DISCLOSURE: This lab contains the use of mechanical and electrical elements that may cause injury if used in an improper manner. Be sure to use all equipment in a dry environment and ensure all digits are clear before tightening any screws or clamps. The overall vacuum system is heavy and should be lifted by two people if needed. Be sure not to lean against the system or to push the system in order to avoid tipping.

Required Materials:

1. KJL Vacuum Trainer
2. Funnel
3. Can of Air
    a. Link

Step 1: Assembly Instructions

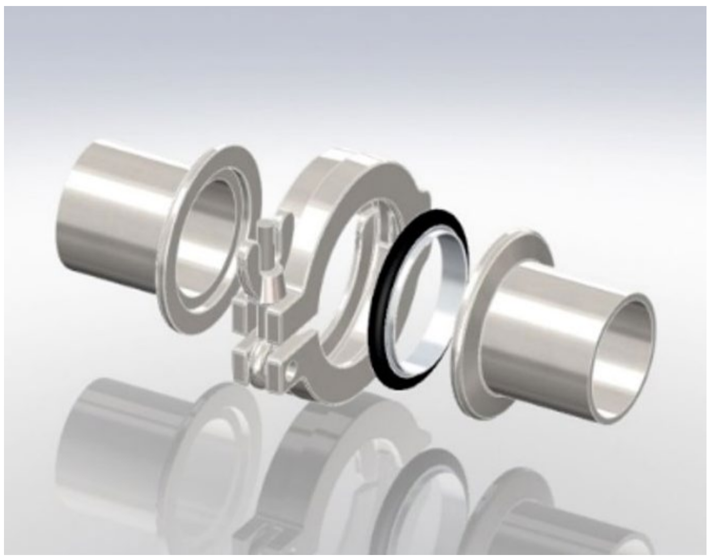


Figure 2.6: Diagram of the assembly of a QF fitting

Figure 2.5: Demonstration of the use of a QF fitting

To assemble the KJL Vacuum trainer, it is important to understand the use of the quick flange (QF) fitting. These fittings are exceedingly simple to use, as seen in the figure. To attach two components together, simply place an O-ring gasket between the two components, and place the flange around the outside. The flange should fit snugly around the flared ends, with a little give if it is squeezed. From here, place the wing nut over the metal platform as seen in the figure, and tighten. Do not overtighten the nut, simply ensure that the screw is hand tight.

Once it is understood how to use a QF fitting, it is time to unpack the KJL Vaccuum Trainer. Altogether, the materials included should be the following:

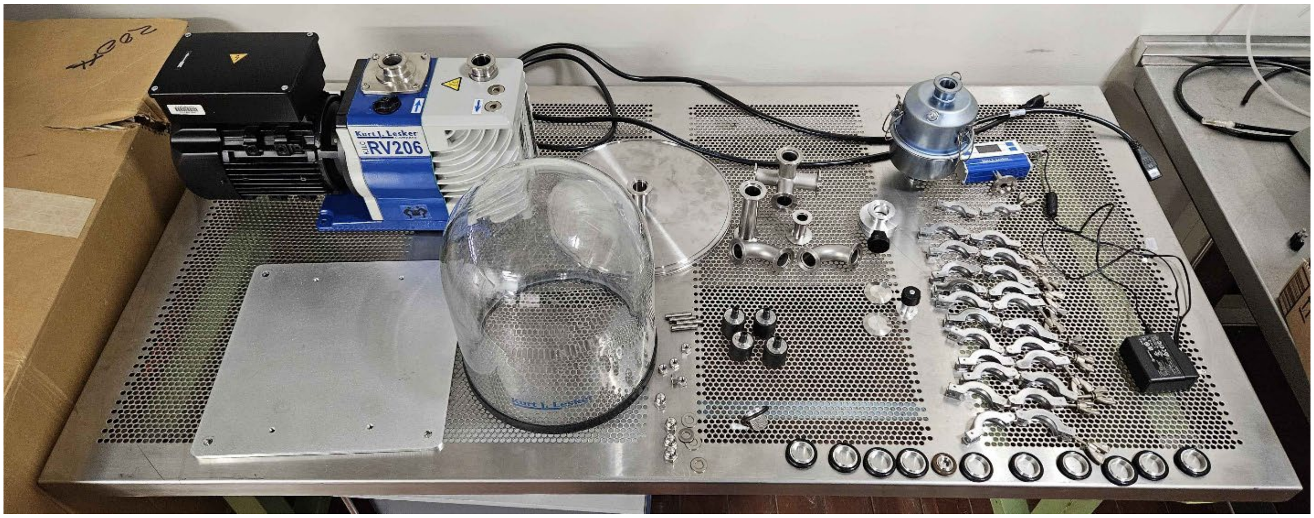
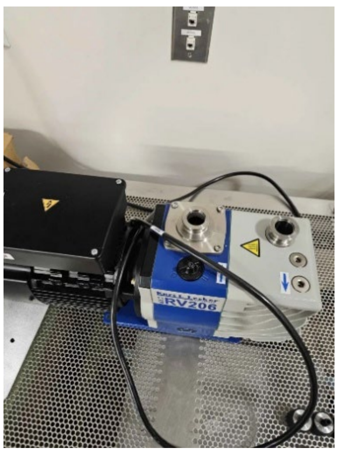


Figure 2.7: Disassembled KJL VTS

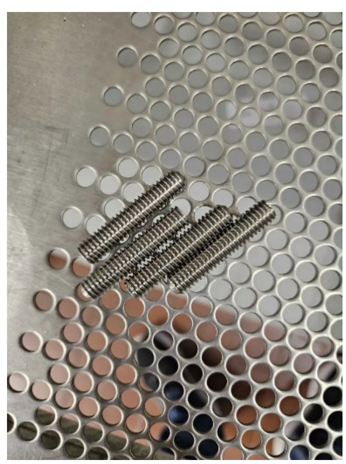


1x Vacuum Pump (With power cable)

1x Baseplate

1x Belljar

1x Pump stand



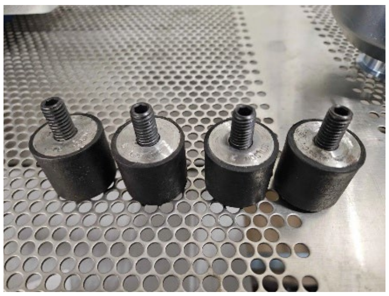
4x Screws

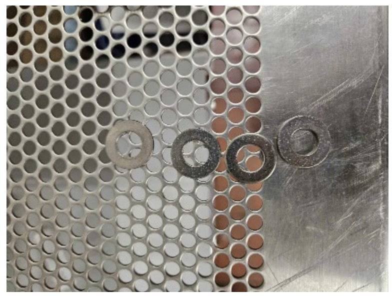
1x 6-way or 4-way (dependent on model purchased) - Will also include an additional 2x blanks, 2x fittings, and 2x o-rings if the model purchased has a 6-way


1x Reducing nozzle


1x Straight


2x Corners


4 x Stand feet


4 x washers


4x top nuts


1x Butterfly valve


1x Vent Valve


1x Exhaust Baffle

1x Vacuum Gauge (With power cable)


8x O-rings and Fittings

1x Bottle of oil

Figure 2.8: Vacuum assembly step 1

Assembling:

Step 1: Filling the pump with oil

Before operating the pump, it is necessary to fill the pump with vacuum oil. Using an Allen key, open the pump as shown in the figure and pour in the oil until the top bar on the gauge is reached. The gauge can be found directly below the Allen key shown in the figure.

Step 2: Assemble the pump stand

First, acquire the required parts. Put each foot into one of the four corners and insert the screws into the central four holes. Ensure that the side with the hex keyhole is on top. Finally, screw the foot nuts onto the feet securely to ensure the feet do not fall out.

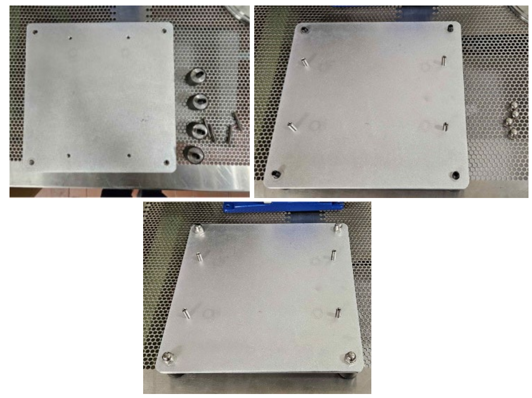


Figure 2.9: Assembling the Pump Stand

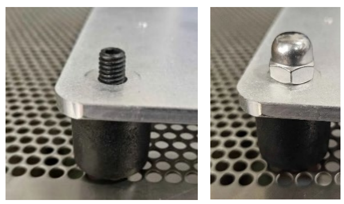


Figure 2.10: Placing the Feet

Step 3: Mount the pump

Place the pump onto the base, with the screws feeding up through the holes in the pump. Now, using the washers and nuts, secure the pump to the base.

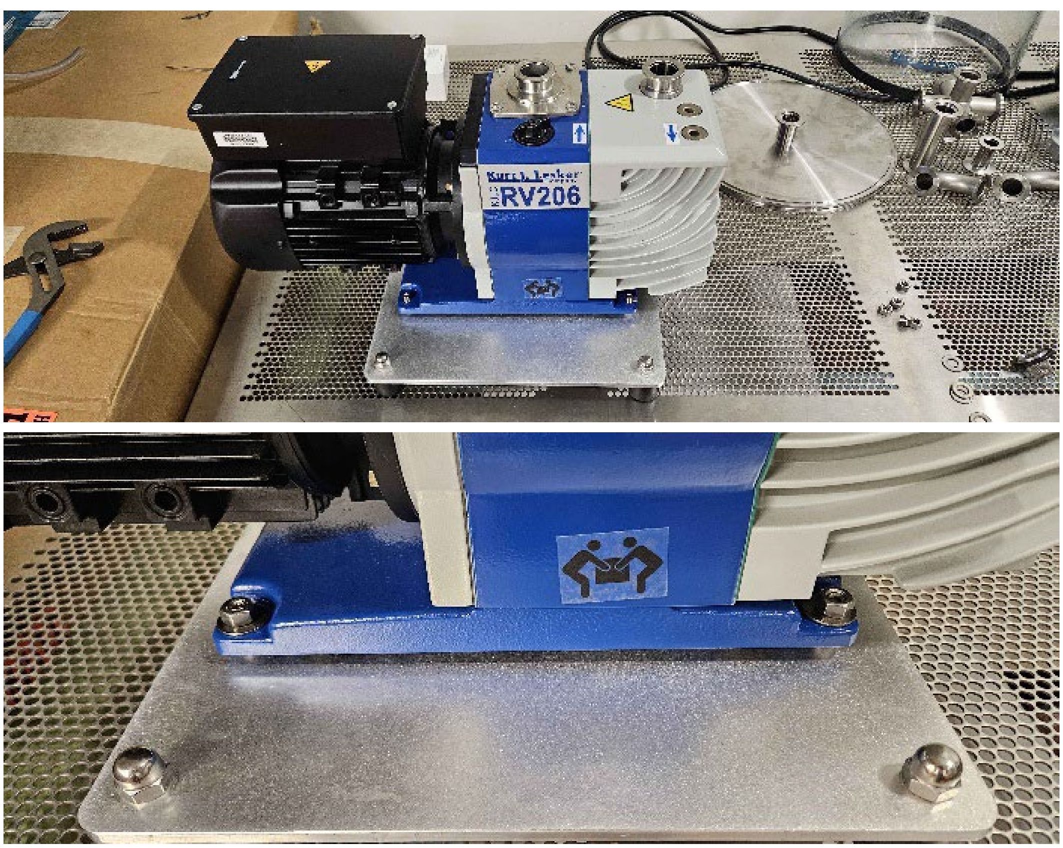


Figure 2.11: Mounting the pump

Step 4: Assemble the vacuum system

First, attach the butterfly valve to the centermost vacuum port, ensuring that the O-ring with the basket is used. The basket should be face down, essentially inside of the centermost port. ***NOTE: When connecting anything directly to the pump, use the shiny QF fittings, as it ensures the best fit.*** Next, grab the 6 or 4-way, and attach it to the top of the butterfly valve. If using the 6-way, place blanks on the left and rear ports. If the 4-way is used, there should be no blanks. Attach the reducing nozzle to the front port if using a 6-way, or the left port if using the 4-way.

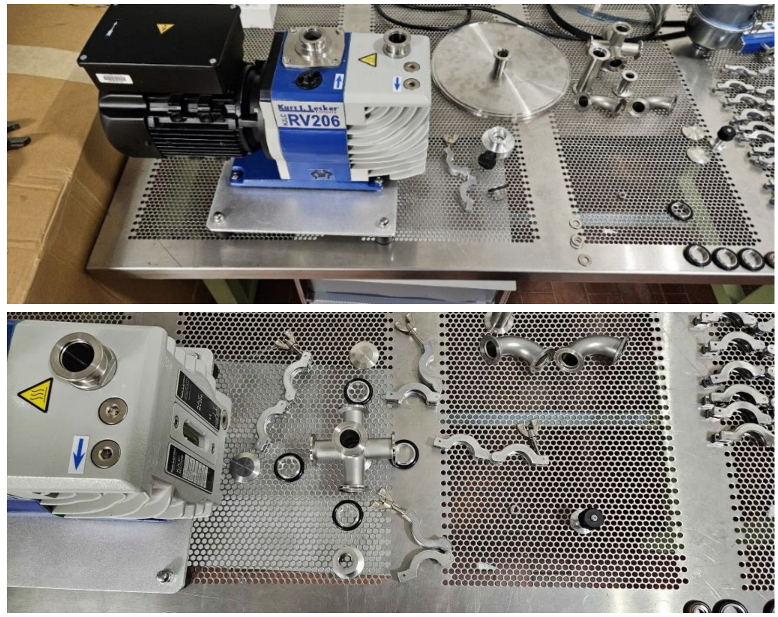


Figure 2.12: Parts required to mount the Butterfly Valve and 6/4 way

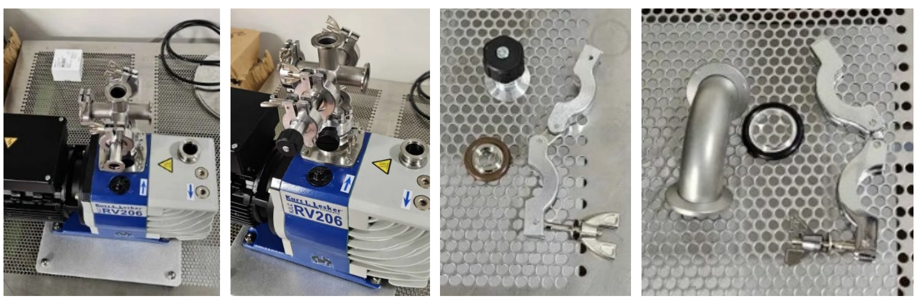


Figure 2.13: Attaching the vent valve

Next, attach the vent valve to the 6 or 4-way, ensuring the O-ring used is oriented properly. Once finished, attach a corner piece to the vacuum exhaust as shown, being sure to use the remaining shiny QF fitting.

Attach the baffle to the corner piece. Ensure that the baffle is oriented properly by connecting the corner piece to the side with the KJL label. If the label has fallen off, one can also confirm the orientation by ensuring it is connected to the side without the clamps. Moving back to the main vacuum system, attach the straight piece and corner to the rightmost port. The corner piece should have its opening facing upwards.

Figure 2.14: Attaching the baffle

Now, attach the baseplate to the top port of the 4 or 6-way. Finally, attach the vacuum gauge to the upwards facing corner piece. Finish the overall assembly by placing the bell jar on the baseplate.

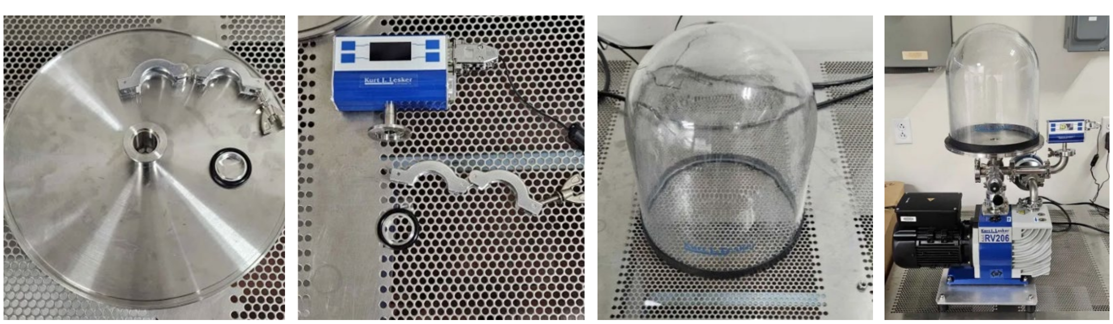


Figure 2.15: Attaching the gauge and baseplate

Step 2: Operating instructions

The overall operation of the KJL Vacuum trainer is very simple. First, ensure that the bell jar is properly placed on the baseplate, meaning that there are no gaps and that the rubber O-ring is seated on top of the circular cut in the baseplate. This will ensure that there are no gaps between the baseplate and the bell jar. To begin pumping, simply turn on the vacuum pump with the respective power switch and open the butterfly valve on the bottom of the system. To tell whether the valve is open or closed, refer to the line on the valve itself. If the line is parallel to the vacuum fittings, then it is open, if it is perpendicular, then it is closed. Allow the system to pump down for some time to achieve what is known as the 'base' pressure, or the lowest pressure the system can achieve. If this is the first time the system was assembled, this value can be as low as 0.01 mTorr, however if the system has been repeatedly assembled, then this value may be higher. The base pressure for the system should be no higher than 20 mTorr, after several reassemblies. If the base pressure is higher than this value before this point, there may be a leak. Refer to the following section for information about this issue. To vent the system, close the butterfly valve and open the small vent valve located on the front or left side of the system, depending on the construction. Be sure to vent the system slowly to avoid damaging the vacuum gauge.

An additional feature of the KJL vacuum trainer that is important to note is the gas ballast. The ballast can be opened or closed depending on the needs of the operator of the pump. For any experiment in this series, and in general, the gas ballast should remain closed as it allows for the lowest possible pressure to be achieved. To confirm that the gas ballast is closed, observe the black switch featured in the figure, and confirm that it is on the C option.

Figure 2.16: Gas Ballast Switch in a Closed Position

Step 3: Troubleshooting Instructions

It is highly likely that the system will not function properly after the first assembly, due to human error. Though, it is possible that some components may be damaged or otherwise ineffective at their roles. This is to be expected, as even in industry, sometimes equipment arrives in a less-than-optimal state. It is up to the technicians and installers to troubleshoot the problem, to accurately diagnose and treat the problem effectively.

The most likely issue to face after assembly is a leak. The simplest way to identify any real leaks in the system will be to listen. Any significant leak (pressure not dropping below 100 torr) will make a hissing noise as the atmosphere attempts to rush in and maintain the pressure. However, it is much more likely to have a small, minute leak that prevents the system from dropping below 100 mTorr. If this is the case, then the first point of concern in to confirm that the bell jar is properly seated on the baseplate. If this does not correct the issue, then a fitting is most likely loose. After rechecking and retightening the fittings, if the issue continues to persist, there is most likely a crack, hole, hair, or other debris on an O-ring somewhere within the system. An effective method for locating this O-ring is to take a can of air duster and spray it around the various fittings of the system, waiting a few seconds after each spray to observe a possible spike in pressure. Once the culprit O-ring is found, a simple swap to a new one will rectify the issue. It is possible to 'induce' one of these leaks by placing a hair across the O-ring diagonally (do not cross the open vacuum section, just the outside) and replacing everything as before. If done correctly after initially finding the base pressure of the system, the new base pressure should be around 3-5 mTorr greater than before.

Questions:

1) Did the system work fully the first time? Why or why not?
2) How were leaks located in the assembled system?
3) What are some systems that vacuums are used in nanotechnology? Research and write about at least 2.
4) What type of pump was used in the KJL training system? How does it work?
5) What was the minimum pressure reached by the system (the base pressure)? What could have an influence on this value?
6) Were there any virtual leaks in the system? What could have caused these to occur?
7) Draw a diagram of the assembled system, labeling all valves and connection points.
8) The O-rings used to create the seals in the various connection points around the system require a particular care routine to maintain them so that they do not dry out or crack early. Research this process and describe it here.
9) Why is it important to ensure the system is dry, and does not have any excessive water vapor?
10) What creates the seal in the vacuum pump?

Supplemental Activities:

Get a solid piece of cardboard that will fit inside the bell jar and put a few puddles of glue on it. Place it into the chamber and begin pumping down the system. Observe the puddles growing in size, and how the pressure changes. How is this related to virtual leaks?