N.B. This guide is written largely for use with the Thin-Film Tunneling with Superconductors instructional lab. However, the practices and procedures contained below can be applied to general evaporation tasks, including the required depositions in the Surface Plasmons lab.

The goal of this experiment (Thin-Film Tunneling with Superconductors) is to investigate the I-V characteristics of electrons tunneling from a superconducting metal, through an insulating metal oxide to a normally conducting metal. To accomplish this, you will be depositing thin films using thermal evaporation of metals. For more details about the experiment itself, see the lab manual on the wiki – this guide is simply to aid in the evaporation aspect of the experiment.

Deposition by evaporation works by heating a metal source until it is hot enough for some atoms to evaporate and then condense on a glass slide. The source sits in a tungsten boat, and a high current passes through the boat. This heats up the boat and the metal source. When atoms evaporate, they travel ballistically in all directions because of the high vacuum – there are few gas atoms to collide with. A mask is placed between the source and the slide to create the desired film pattern (shown in Figure 14 -Figure 16). Metal atoms will only condense on the slide in the areas that are not shadowed by the mask. After each pattern is deposited, a different source and mask are moved under the slide to create the next pattern.

![Figure 1. Thermal evaporation.](image)
The evaporator with which you will deposit the metallic films uses a turbo molecular pump. This pump is worth about $30K and can be easily destroyed if it receives a significant bump. Please be careful when working with or around the evaporator. Do not operate the evaporator without first receiving personal instruction from one of the lab staff or TAs. To keep the chamber clean, always keep it under vacuum when not in use.

![Evaporator](image)

Figure 2. Evaporator.

Make sure the slides are extremely clean. As delivered the slides should be sufficiently clean, but touching them with ungloved hands, even just by the edges, will make the slides dirty. Unfortunately, most slides have been contaminated and chances are you will have to wash one. Clean it very carefully with a series of washings in acetone and methyl alcohol. Pour about ¼” of acetone from the large brown bottle into a clean glass beaker. Do the same with alcohol in another clean glass beaker. Do not use the solvents from the plastic squeeze bottles. The acetone and alcohol you use must be very pure, since any contamination present tends to leave a filmy residue.

Place a slide in the acetone and agitate the beaker by putting it in the sonicator. The sonicator should have a small amount of tap water in it (about an inch); simply place the beaker on the bottom of the sonicator and turn it on. Let it run for a couple of minutes. Using tongs pick up the slide by one corner and let the acetone drain off. Place the slide in the alcohol and repeat the process. You should go back and forth between the acetone and alcohol several times until you
are convinced the slide is clean. Between each step, take a second to let the liquid drop off into its own beaker to avoid mixing the acetone and alcohol.
If this process does not clean the slide enough, you can make an Alconox solution with distilled water to wash the slide before using acetone and alcohol. Always use alcohol last. Let the alcohol drip off and then gently dab the bottom corner on a Kimwipe to make the last drop fall off.
After the last alcohol cleaning use the tongs to place the slide in the desiccator containing calcium chloride. Position the slide with one corner down making as little contact with other objects as possible. Place the lid on the desiccator and wait for the slide to dry.

To ensure that the desired vacuum level is reached quickly, the cold trap must be filled with liquid nitrogen. You will be trained in proper safety procedures for handling liquid nitrogen. Pour the nitrogen into the nitrogen cup as seen in Figure 3. It must be poured quickly enough that liquid accumulates in the cup. If poured too slowly it will just boil off upon contact. Continue to pour nitrogen until the hole leading to the chamber begins to overflow. Repeat this process once after 15-30 minutes to ensure a full trap.

![Figure 3. Nitrogen cup and shutter (in blocking position)](image)

You will need to prepare all the materials in the evaporator before you begin. This includes placing the materials to be evaporated in boats suspended between the connection terminals, putting the shields in place (and making sure they don’t touch the “hot” post on the right,) and putting the masks in place. Make sure the boats and masks are properly aligned and that the connection terminals are tight. See the TAs for access to the materials to be evaporated and the boats to hold them. It may be the case that some boats were left in the evaporator from a previous evaporation; be sure to replace the chromium and silver boats. Check the lead boat; if there is still lead in the boat, you may reuse it (so little lead is used in the evaporation, it is often good for several evaporations). **Make sure the liquid nitrogen trap on the evaporator system is full before any evaporations are performed.**
The way to ensure success, of course, is to be very careful about the cleanliness of the vacuum chamber. DO NOT use vacuum grease on the evaporator/vacuum system. Use a Kimwipe and alcohol to wipe down the chamber door and O-ring every time the door is opened. When putting your hands in the chamber, always wear gloves.

Place the metals into their positions between the connection terminals. The chromium is evaporated from a bar, half a pellet of aluminum is placed in a tungsten basket, and three pellets of lead are placed in a tungsten boat (Figure 5). Place these holders between the posts, only in the spot indicated for each metal (e.g. put the aluminum in the position marked Al). To make a good contact, screw them tightly into the posts underneath the metal washer. To ensure proper alignment with the masks, the chromium bar and aluminum basket must be placed so that the left end is in front of the left post, and the right end is behind the right post, as seen in Figure 6. The aluminum basket must have the top wire on the left, as seen in Figure 7. Check that the masks are in the appropriate positions.

Check that the shield (Figure 4) can be placed over the metal without touching the holder, and that the holder does not touch the shield or the ring underneath; this could cause a short that would prevent current from passing through the bar or boat.

Remove the shield and place the metal in the boat.

Put the shield down again, making sure the element’s name faces towards you.

Place your now cleaned and dry slide in the rotating holder (above the masks). Once the slide is in place, move the sample arm back into the center of the chamber, as far as it will go, then pull it towards you. It should stop after a small motion. The sample is now properly oriented relative to the mask (see Correct Figure 8).

![Figure 4. Chromium shield inside evaporation chamber](image)

![Figure 5. Left: Chromium rod. Center: Tungsten basket for aluminum. Right: Tungsten boat for lead.](image)
Once the metals, masks, and shields are in place, you are ready to pump down the vacuum chamber of the evaporator. Close the evaporator door, press “cycle” on the vacuum controller (Figure 9) and wait a few minutes for the pumping to finish. Once the chamber pressure reaches about $5 \times 10^{-6}$ Torr, you can begin the evaporation process. Remember to proceed in the correct order (Cr, Al, oxidation, Pb). Make sure the liquid nitrogen trap on the evaporator system is full before any evaporations are performed.
Figure 9. Thickness monitor and vacuum controller.

Figure 10. Source selection dial.

Turn the source selection dial (Figure 10) so that source 1 (Cr) is on the side marked “Evaporate”. Look through the side window to ensure that the post on which the boat rests fits snugly against the contact (also called the “brush”). This will ensure that the mask is properly aligned relative to the slide, and a strong, constant current flows through the boat. See Figure 11 and Figure 12 for correct and incorrect positioning.
Figure 11. Correct positioning of evaporation terminal against contact (brush). (Top view, view through side window.)

Figure 12. Incorrect positioning of evaporation terminal against contact brush. (Top view, view through side window.)

You will monitor the thickness of your evaporations with a thickness monitor (Figure 9). Before performing the evaporations, ensure that the thickness monitor is set to the correct densities and Z-factors for the materials you will be evaporating. These are listed on the evaporator control panel.

Before performing evaporations, you will want to burn off any impurities in the metal by putting the evaporator’s shutter into the “blocking” position (see Figure 3) and setting the current slightly (0.5-1 A) below the nominal melting currents marked on the evaporator for ~30s by adjusting the power supply (Figure 13). Set the thickness monitor to the appropriate metal. You will then move the shutter to the “open” position and increase the current to just above the nominal melting value, and “zero” the deposition monitor. The deposition rate should increase to a non-zero value. Adjust the current to achieve the desired deposition rate. A faster rate results in a lower-quality film, so choose your deposition rate to balance time and quality. Wait ~30s after changing the current for the metal to heat up to see the effect on the deposition rate. When the desired film thickness is reached, close the shutter, then move to the next evaporation source.
Chromium pads should be deposited first as pictured in Figure 14. Chromium is used to make electrical contacts because it forms a hard film which is scratch resistant. Make the Cr contacts about 1-2 kÅ thick at a deposition rate of about 2-10 Å per second.

Aluminum (Al) is used to make the single long strip down the center of the slide. This aluminum strip will be the normally conducting metal. Use a deposition rate of 5-20 Å/sec to achieve a final thickness of 800 - 1800 Å. Al films are continuous after 200 Angstroms – the device will function as needed as long as you exceed this thickness. Note: The heat generated during evaporation tends to warm the substrate. If the bell jar is vented before the substrate is cool enough, then the metal film undergoes rapid oxidation, creating a “fog” on the surface of the metal.

You will create the insulating oxide layer on top of the Al (creating aluminum oxide, Al₂O₃) by venting the system back to atmospheric pressure (let the sample cool before venting and leave the door closed!) and leaving it there for 5-30 minutes. 10 minutes is a good starting time but lower humidity may require a longer oxidation time, and vice versa. Be sure to let the sample cool before venting to prevent fogging. Afterwards, pump the chamber back down and proceed to the Pb evaporation.
Figure 15. Aluminum strip.

Figure 16. Lead strips.

Lead (Pb) is used to make the three short strips that run across the slide. The lead strips will be the superconducting metal. Be aware that lead melts at a very low temperature compared to the other metals used. It is relatively easy to evaporate all of it while attempting to burn off impurities. Deposition rates of 5-20 Å/sec or even a little higher are fine. Final depositions of 800 - 1800 Å are typical. The Pb film will be continuous once you have reached ~700 Å. The metal for this experiment must be clean, free of fingerprints (both below and on the surface) and have a mirror-like finish.

When finished, turn the current down to zero, but do not turn the power supply off. Press “vent” (Figure 9), and when the pressure reaches atmosphere, unscrew the lock and open the door. It may be difficult to pull open but do not be afraid to pull hard, as long as the pressure is $7.60 \times 10^2$ Torr.

Now with a completed slide, you can continue the experiment as in the lab manual.

Be sure to look below at the supplementary images to get a better familiarity with the evaporator.
Supplementary images:

Figure 17. Shutter open.

Figure 18. Shutter blocking.
Figure 19. Mask misaligned due to improper position of post relative to contact brush.