

Sample Preparation procedure for the VLSPGM Beamline

The following is a guideline on how to prepare samples using the provided sample holders.

The BL staff at the beginning of your beamtime will give you:

- 6 sample holders that have been previously thoroughly cleaned using the procedure in Appendix A
- A roll of double sided carbon tape
- Clean lab tools (e.g. tweezers, spatula, scissors, mortar & pestle)

In the assigned lab, and workstation, you will find Kim-wipes, gloves, beakers, solvents (acetone and methanol) and a can of compressed air.

If any of the material is missing, please ask.

Sample preparation

1. Take a clean sample holder and place it on a clean large Kim-wipe.

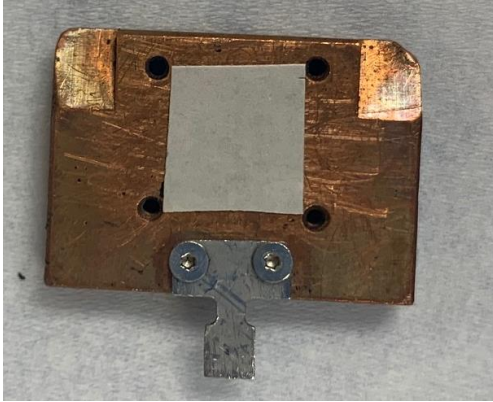
The Kim-wipe will be thrown in the garbage once the sample holder has been prepared, avoiding sample's spilling to contaminate subsequent sample holders.

2. Cut one piece of double sided carbon tape and tape it on the sample holder, [Picture 1](#).

The size of the sample must not be bigger than the area between the 4 screw-holes on the holder.

Indicatively, if the sample is a solid piece or has been prepared in a pellet form, the size must not be larger than 0.8cmx0.8cm. If the sample is powder, it must not be spread anywhere outside the 4 screw-holes area.

If the sample is a solid piece (e.g. steel plate for tribology sample), it cannot be thicker than 4.5mm.

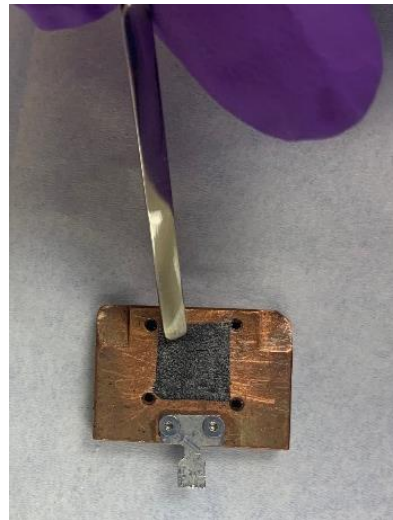
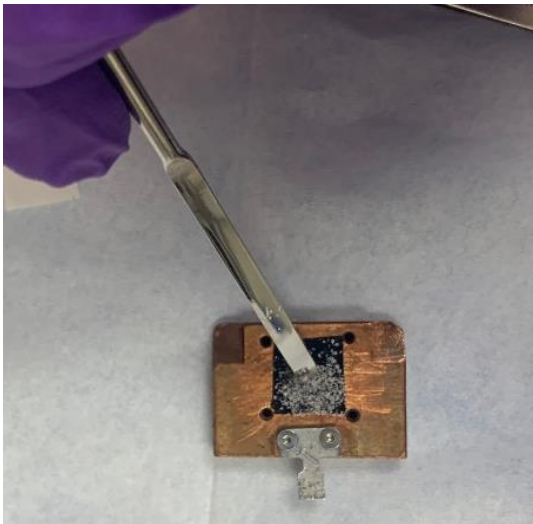


Picture 1: Sample older with attached the double-sided carbon tape, this is the maximum allowed size of C-tape. It can be smaller. It CANNOT be bigger.

3. Attach the sample onto the carbon tape. If it is powder, with a clean spatula place a small amount on the tape and smear it as uniform as possible to form a very thin layer covering the full tape, Picture 2.

If the powder sample is coarse, use clean mortar and pestle to grind it into a finest powder.

Remember to thoroughly clean the tools you used before moving to a new sample, wiping with several clean acetone soaked Kim-wipes and to replace the large Kim-wipe.



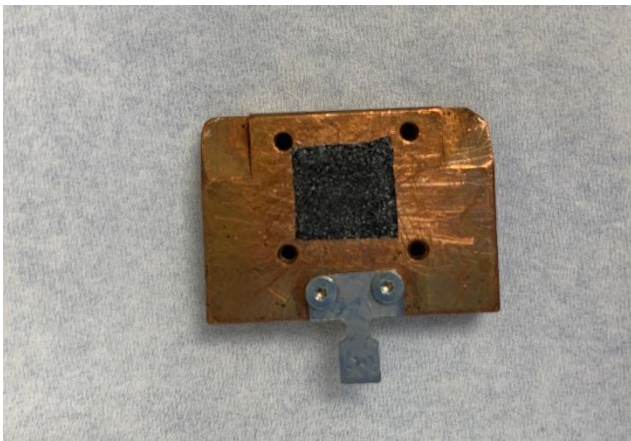
Picture 2: Powder samples on carbon tape

3. Use the compressed air can to gently remove the exceeding sample powder not glue to the tape.

This action prevents the loose powder to float in the experimental chamber, contaminating the chamber and detector's surfaces and worsening the vacuum. Do not old the sample above other samples when spraying the air to avoid powder to deposit on top of clean tools, clean sample holders or already prepared sample.

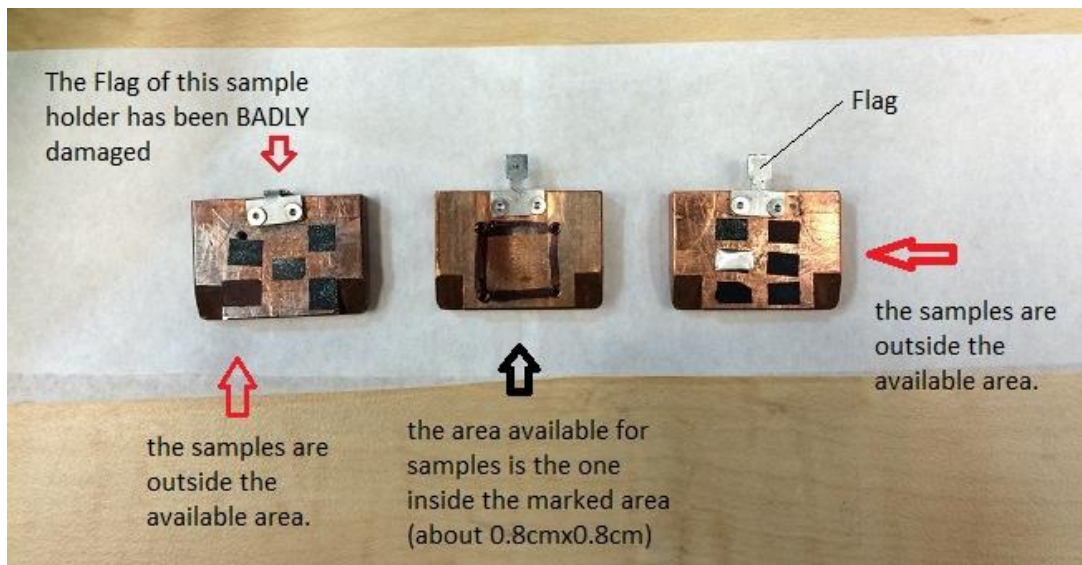
Be shrewd.

An example of how your sample should look like is shown in Picture 3.



Picture 3: Nicely prepared sample – how it should look like 😊

Picture 4 shows how your sample should NOT look like.

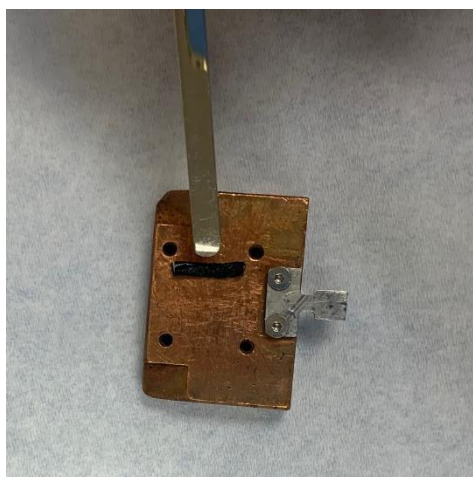


Picture 4: How NOT to mount samples 😞

Remember to always thoroughly clean the tools you used before moving to a new sample wiping them with several clean acetone soaked Kim-wipes and to replace the large Kim-wipe.

Cleaning the sample holder:

- a. To remove the “tape+sample” from the holder, after that particular sample has been analysed, use the spatula or the tweezers to first remove the carbon tape, Picture 5.



Picture 5: Removing of the tape

- b. Wipe the sample holder with an acetone soaked Kim-wipe
- c. Repeat (b)
- d. Wipe the sample holder with a methanol soaked Kim-wipe
- e. Repeat (d)

The procedure should completely remove any traces of carbon tape and glue from the holder.

Remember that the tape's glue presents traces of chemicals (e.g. Boron) that could interfere with your results.

Appendix A

Occasionally during the beamtime is necessary to thoroughly clean the sample holder. This is the procedure followed by the BL staff between every Users group.

- i. Place the sample holder in an acetone filled beaker, just enough to fully submerge the holder, Picture 6



Picture 6: Beaker with an acetone fully submerged sample holder

- ii. Place the beaker in the ultrasonic cleaner, switched it on setting the timer to 10 minutes, Picture 7.

Remember the ultrasonic cleaner has to be fill with some water before switch it on. If you never used one, ask for help.



Picture 7: Ultrasonic cleaner with some water and the beaker containing the sample holder and the acetone

iii. Take the sample holder out from the beaker, rinse with acetone, wipe it dry

The sample holder is now clean and ready to be used.