SEM Sample Preparation Instructions

1. Sample Cleaning and Drying:

Before SEM characterization, samples must be thoroughly degreased and dried to eliminate any outgasing from organic contamination and water. Samples can be cleaned ultrasonically using solvent like acetone and methanol (or alcohol). When cleaned by volatile solvent, samples can be blown dry using a compressed gas. After cleaned by water, samples should be dried completely using oven or hot plate. Surface dusts are removed using above processes, and they can also be removed by blowing a compressed gas.

2. Handling:

Gloves must be worn all the time during sample preparation and transfer. Do not touch samples, sample holders, sample stubs, and sample exchange tool with bare hands. Grease from hand is the major contamination to the SEM system. All parts and tools should be handled on a clean paper like Kimwipes or similar.

3. Sample mounting:

Normally samples are mounted on the holders or stubs using double-sided conductive tapes. **Only** use the vacuum compatible carbon and copper tapes provided by SEM lab. You can also use silver paint for sample mounting with complete drying before loading into SEM chamber. Small cylinder-like samples may be mounted directly on pore-plate.

4. Powder samples:

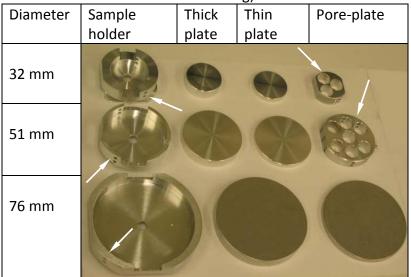
Powder materials are hard to mount and need special handling for sample mounting. Otherwise they may get loose and fly off the holder in the vacuum and under the beam. For small amount of powder, they can be dispersed in a volatile solvent like Acetone and alcohol, and then drops of the mixture can be dripped onto a clean substrate. After drying the powder particles should be dispersed onto the substrate surface. For large amount of powder, they can be compressed into small disks for sample mounting. Carbon, copper tapes can be used for powder mounting. Sprinkle the powder lightly with a spatula, press lightly to seat. Turn the sample holder upside down and tap it to remove loose material.

5. Less conductive samples:

When samples are not very conductive, charge effect will cause image distortion or drift. Low acceleration voltage should be used to reduce the charge effect if samples cannot be coated with a conductive coating. To complete eliminate the charge effect, samples should be coated. SEM lab has sputtering coater for gold coating. The coating thickness could be several nanometer to tens nanometer depending on if the coating interfere with the morphology of your sample. After coating, the sample should be mounted with a conductive "bridge" (e.g. carbon / copper tapes, or silver paint) connected from the top surface of the sample to the sample holder.

- 6. Do not take any tools, consumables away from the SEM lab. Always replace them to the original position, and keep the working space organized and clean.
- 7. Please don't expose the vacuum chamber to air for a long time. Otherwise, it increases the contamination inside and cause carbon deposition on your sample during the imaging. And when the EDS detector is cooled by liquid nitrogen, ice will form on detector and affect its performance. If it takes a while for you to get your samples ready, then vent the chamber only when your samples are ready.

8. There are three different sample holders, each holder have 2-3 different sample plates as shown below. Choose the right holder and plate according to your sample size. (White arrows indicate set screws for mounting)



- For the pore-plate, there are two types of stubs: 12.5mm diameter × 5mm height, and 12.5mm diameter × 10mm height.
- 10. Samples can be attached to the flat plates using double-side carbon or copper tapes. Small samples can also be attached onto stubs and then mounted to pore-plates using setscrews.
- 11. Sample plates should be seated in the corresponding sample holder securely, and fastened with setscrews.

Note: Do not loosen the setscrews too much. It is easy to get them lost.

- 12. Using the sample exchange tool to transfer the sample holder into or out of the sample chamber. When load the sample holder onto the exchange tool, the flat edge of the holder should point outward as shown in figure below.
- 13. Press the release buttons of the sample exchange tool, and then remove the holder from the hook.

