Sample Preparation

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 such as isopropanol, methanol and acetone. 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.

Sample 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.

Sample Mounting

Normally samples are mounted on the holders or stubs using double-sided conductive tapes. Only use the vacuum compatible carbon 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.

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 and the 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.

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 completely eliminate the charge effect, samples should be coated using a sputtering coater. 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.

EDS Sample Preparation

There is no special sample preparation for EDS, other than that required to image the sample in the SEM, is required for qualitative analysis, but for quantitative analysis in the SEM the sample must be bulk, flat and polished.