Sample Preparation for Powders for XPS Analysis

When using powders, proper sample preparation is key to keeping the instrument clean and free from contamination.

1. The ideal situation for examining loose powders is to press the powder into high purity indium foil. The indium foil needs to be high purity (thus expensive) and the powder needs to be compatible with indium.
2. If this is not possible then dissolving the powder and making a thin film and coating an appropriate substrate would be the next best thing.
3. If the above is impossible, then loose powder can be used as long as proper precautions are taken. Carbon tape for SEM is the most commonly used tape but other double tape that is low outgassing is OK. If your sample is conducting and you use a nonconducting tape you will have sample charging problems. Follow the procedure below.

Procedure for use of Loose Powders in XPS

Materials needed:

Spatula of appropriate size for powder container	Isopropyl alcohol
Flat sided spatula for pressing powder	Kim wipes
Double sided tape or Carbon tape	Aluminum foil
Clean scissors	Pipette bulb (if needed)

Procedure for Mounting Loose Powders

a. Put on a new pair of powder free gloves.
b. Prepare a clean work area, cover with large piece of fresh clean room wipe and aluminum foil.
c. Clean your puck or sample holder with IPA.
d. Place the clean sample holder onto aluminum foil.
e. Cut small pieces of tape, 5 x 5 mm or smaller (1 small piece for each sample).
f. Place a piece of tape down on the puck or stub with the paper on the top side.
g. If using double-sided tape with protective paper, you can place all your tape pieces on the stube or puck, otherwise if using double-sided tap that is bare on top only place one piece at a time. Space the tape pieces out quite well to avoid cross contamination between the samples.
h. Remove the top covering on one piece of tape.
i. Use your spatula and place a small amount of powder onto the tap. You do not want a thick layer, but you want to completely cover the tape.
j. Spread the powder so it covers as much of the tape as possible – Try to ensure that you have an area that “appears” completely covered with no tape showing through.

k. Use the flat side of a scoopula or spatula to push your powder sample firmly down into the tape.
l. Tip the sample holder over to remove loose powder onto you Al foil. **Do not contaminate the bench, keep all the powder contained on your wipe and Al foi**l. (Alternatively, use a pipette bulb or other small bulb to **gently** blow at the surface of the powder to remove any unbound powder. Blow the sample onto the Al foil (do not contaminate the bench). Blow away from other samples that have already been placed on the holder. The best option is to direct the blowing from the bulb off the holder
m. Repeat steps f-l for as many samples as you have. Ensure all loose powder is removed from the surface of the holder.
n. Turn the puck or Stub completely over and shake any loose powder off.
o. Check that the powder still on the tape is well attached to the tape.
p. Place the sample into the load lock.
q. Dispose of the Al foil and wipe. Put a new wipe down on the bench.