CMMP X-ray Photo-electron Spectroscopy

Wednesday, August 14, 2013

Mounting Powder Samples for XPS

There are several methods for mounting powder samples for XPS. One is to press the powder into a very thin metal foil that is malleable enough to allow the powder particles to impregnate the foil. Indium is the most common choice, and in this case the sample powder is rolled or pressed into the indium foil and that powder-impregnated foil is used as the XPS sample. Since no adhesive is used, this is a truly "ultra high vacuum" method of sample mounting. One problem with this method is that indium, being a fairly high-Z material, can provide significant spectral contamination from its many XPS and Auger peaks.

To remedy the complication of pressing samples into metal foils, as well as the corresponding spectral contamination, we have successfully used double-stick carbon tape used in SEM sample preparation. The mounting procedure is shown below:

Step 1: Place a disk of double-stick carbon tape on XPS stub. It should be centered on the stub so that the ion-gun rastering can be easily reduced to facilitate sputtering just the sample, thus increasing the ion-density and thus the sputtering yield.

Since the carbon-tape disks will be introduced into an UHV environment, it is best to keep the disks in a dry environment such as a desiccator. The disk pictured is roughly 1 cm in diameter. Rub and press the adhesive disk onto the XPS stub. If the XPS is coated, the adhesive disk might not stick as it may remove the stub coating. This is acceptable. Let the adhesive disk remove the coating-- just replace the adhesive disk where the film has been removed from the XPS stub.

Step 2: Using a spatula, place the powder on the exposed adhesive disk. Again, since XPS requires a UHV environment, the powder needs to be appropriate for that environment. If the sample is inherently porous, it should be baked out under vacuum to remove as much physisorbed water and solvent as possible. After baking under vacuum such powders should be stored in vacuum or in a desiccator. As this is a UHV technique, manipulate the powder only with UHV clean spatulas.

Some materials simply have very poor vacuum characteristics. Use the ion-gauge and residual gas analyzer look for signs of problems before introducing the sample

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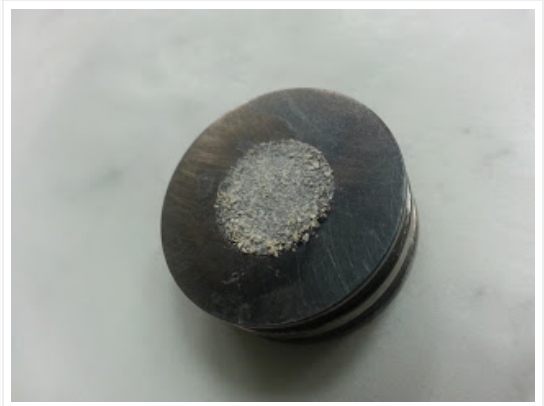
into the main XPS analysis chamber. Is the base pressure too high? Has the sample visibly changed appearance? If a problem is suspected, the RGA will give a fingerprint of the intro-chamber vacuum environment. If there is a huge background of water or organics-- do not introduce the sample into the XPS.

Step 3: A pile of powder will not make it into the the XPS safely. It should be noted that powders migrating through the XPS vacuum system is potentially very destructive to the instrument. Migrating powder can land on valve sealing surfaces causing them to leak and damage them so that they can never seal. Migrating powders can also destroy the turbo-molecular pump and the mechanical pump that backs it.

To properly prepare the powder sample, use a spatula to smear the powder across the surface of the adhesive disk of carbon tape. Distribute the powder as evenly as possible and uniformly cover the carbon adhesive. A failure to do so will result in significant C 1s, and to a smaller extent O 1s, XPS background from the tape. Note that the stub shown in the third image can not and should not be placed in the XPS intro-chamber. The unattached powder can migrate into the vacuum system from the laminar flow during pump-down as well as through the vibration of pneumatic valves opening and closing. By "damage" I don't mean some down time and an interruption of the work flow-- I mean that plus as much as $20,000 of damage.



To get the stub ready for the XPS, tap the stub very hard on a rigid surface. Do this until there is no evidence of any powder coming off the surface. Tapping on a white weight paper can help. While it may seem that a good deal of powder has fallen off-- that is acceptable. The XPS is a surface technique that looks at a few nano-meters of the sample surface. A fine dusting of material, seemingly insignificant to the naked eye, is sufficient. If your sample is multiphase and one component is a fine powder while other is larger chunks-- then this can

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About Me

Dr. Eric Lochner

I manage the analytical and fabrication tools in the Condensed Matter and Materials Physics (CMMP) group at Florida State University. Please contact me directly at lochner@physics.fsu.edu for information about using any of these research tools.

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lead to some sampling bias, so consider this in sample preparation.

Posted by Dr. Eric Lochner at 10:55 AM

Labels: base pressure, powder, RGA, XPS basics

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Nina Athena January 23, 2020 at 6:57 PM

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