

Sample Preparation Guidelines

Materials Bay

Scanning Electron Microscopy (SEM)

Samples prepared for applications involving the SEM should be clean, dry and must not outgas. The guidelines that must be followed are as follows:

- a. Thin/Bulk films: 1-day desiccation
 - Plasma cleaner to be used for surface cleaning.
- b. Powder samples: 2 days desiccation
- c. Polymer: 3 days desiccation
 - Prior approval of the polymer sample by the tool owner, is required. Also, polymers/photoresist samples are *allowed only during the day* (so that FTs can monitor).
 - Complete degassing of samples is needed. For facilitating degassing of samples outside the SEM tool, the samples should be under desiccation for a minimum of 3 days with silica gel to absorb the moisture/humidity. Alternatively, samples can be subjected to suitable heating on a hot plate/oven e.g. 3 minutes at 110 °C on a hot plate, for positive photoresists.

Note:

- a. For imaging in the SEM, we suggest depositing a couple of nm of gold on non-conducting samples.
- b. For imaging and determining the distribution of particles in powder-samples we suggest drop casting of the sample on any conducting substrate.
- c. For EDS and Mono CL analysis, we suggest plasma cleaning of samples prior to the analysis.
- d. Imaging and measurements on wet samples, as-is, is not possible, but, if desirable, these samples can be drop-cast onto a conducting substrate and after solvent evaporation (and desiccation), subjected to analysis in the SEM.

Focused Ion Beam (FIB)

Generic Samples

Samples prepared for FIB should be clean, dry and must not outgas. The guidelines that must be followed are as follows:

- a. Thin/Bulk films: 1-day desiccation
 - Plasma cleaner to be used for surface cleaning
- b. Powder samples: 2 days desiccation
- c. Polymer: 3 days desiccation
 - Prior approval of the polymer sample by the tool owner, is required. Also, polymers/photoresist samples are *allowed only during the day* (so that FTs can monitor).
 - Complete degassing of samples is needed. For facilitating degassing of samples outside the FIB tool, the samples should be under desiccation for a minimum of 3 days with silica gel to absorb the moisture/humidity. Alternatively, samples can be subjected to suitable heating on a hot plate/oven e.g. 3 minutes at 110 °C on a hot plate, for positive photoresists.
- d. Maximum sample size is 75 mm/ 3 inch.

Transmission Electron Microscopy (TEM) samples

- a. Users are requested to collect the TEM grids from the tool owner one day prior to the slot.

X-ray Photoelectron Spectroscopy (XPS)

Samples prepared for applications involving the XPS system should be extremely clean, dry and must not outgas. The guidelines that must be followed are as follows:

- a. Thin/Bulk films:
 - The samples must be flat and free from contamination. Biological samples of living or dead tissue or material derived from plants/ animals are not permitted.
 - Always use a clean pair of forceps to handle samples.
 - Use only the vacuum-compatible carbon tape provided.
 - Do not use scotch tape / cello-tape / glue.
 - The sample must be dried completely in desiccators under vacuum with appropriate desiccant, at least for one day before loading the sample.
- b. Powder samples:
 - Method 1: Press powders into pellet (thin) form and then load into the XPS system. (Highly recommended).
 - Method 2: Suspend powders in a suitable solvent and then drop a small quantity of the suspension over a small piece (5 mm X 5 mm) of cleaned silicon wafer. Dry the wafer thoroughly in air and then in a desiccator under vacuum for at least 48 hours.
 - Method 3: Paste a small piece of carbon tape on a Si wafer/conducting substrate and drop the powder onto the tape. Keep the sample in the desiccator under vacuum for at least 24 hours.
- c. Polymer:
 - Speak to the concerned FT/SFT.

Transmission Electron Microscopy (TEM)

- We will allow most kinds of non-magnetic specimens such as metals, semiconductor samples, powder samples, solution-based nano-particle samples. But, the samples have to be high-vacuum compatible.
- The samples have to be in 3 mm diameter, and thickness, preferably less than 100 nm (for HRTEM preferred sample thickness is less than 50 nm). The powder and solution based nano-particle samples have to be dispersed well in a suitable solvent and drop cast on the TEM grid of copper or molybdenum.
- We will not entertain any kind of **biological samples** and **magnetic bulk sample**, however, exceptions will be allowed on a case by case basis.
- The prepared TEM samples need to be dry and free of contamination. The samples can be brought with the user (in a desiccator) at the time of analysis. However, the user has to make sure he/ she takes back the sample after the analysis as no user-samples will be retained or stored in the TEM facility.
- Powder/ solution based samples on copper grids have to be desiccated for at least 10 hours. At the time of equipment usage, if the samples have significant moisture, the user will not be allowed to use the TEM.
- The samples have to be prepared by the users themselves, but, the staff in charge will be there to guide you in the sample preparation for different kinds of samples.

Electrical Bay

- For Hall measurements, bulk samples should be 1 cm x 1 cm laterally (thickness, up to a few mm), with silver paint contacts made on edges.
- For all IV/CV measurements on semiconductor chips with bottom contacts, the back side of the chips should have Al metal contacts. Further, these contacts should be bonded to a substrate (e.g. a piece of bare Si wafer that has a layer of Al on top, that is larger than the device) using silver paste in such a way that the chip area is smaller than the Al area on top

of the substrate. For CV measurements on pellets, metal contacts should be made to the top and bottom surface of the pellets.

- For RF measurements, samples should have GSG (Ground-Signal-Ground) pattern. The distance between probe pads should be 100 μm or 150 μm or 200 μm .

Optical Bay

UV VIS Spectrophotometer

1. Thin Films samples
 - Sample size should be at least 2 cm x 2 cm
 - Users must bring the substrate for baseline correction
2. Solutions samples
 - Users must bring the solvent for baseline correction
 - Sample concentration should be on the order of 0.01M.
3. Powder samples
 - Powder sample quantity should be on the order of gm.

Zeta Phase Analysis Light Scattering (Zeta PALS)

For Zeta Potential Measurements:

- Sample volume: 1250 μL
- Sample concentration: 0.1 mg/mL to 1mg/mL

For Particle Size Measurements:

- Sample volume: 1 to 3 mL
- Concentration range: 0.1 mg/mL

Fourier Transform Infrared Spectroscopy (FTIR)

- For thin film structures, for analysis in reflection mode, minimum sample size is 1 cm x 1cm (maximum size of the sample: 5 cm x 5 cm).
- Please clean the pellet dies and related accessories after use, else it will lead to corrosion.
- Always use gloves while using FTIR and accessories.
- Use KBr powder sensibly.
- Do not use samples containing water in liquid/gas cell or any other KBr window.

Simultaneous Thermal Analyzer (STA)

- Make sure chiller is at 20 $^{\circ}\text{C}$ and purge gas is flowing through the furnace.
- Do not open the furnace lid if the furnace temperature is above 50 $^{\circ}\text{C}$

X-ray Diffraction (XRD)

Samples with flat surface is preferred.

Mechanical Bay

Atomic Force Microscopy (AFM)

- Sample surface should be flat, clean and dry.
- For thickness measurement of films, we need a reference substrate side (uncoated region).
- For modes like KPFM, C-AFM, PFM samples should be conductive.

Optical Profilometer (OP)

- The samples should be reflective.
- For thickness measurement of films, we need a reference substrate side (uncoated region).

Micro System Analyzer (MSA)

- The Out of Plane devices should be reflective enough.

Micro UTM

- Samples preparation for tensile, compression and bending to be prepared according to prescribed ASTM Standards specific to types of samples and tests.

Scanning Acoustic Microscope (SAM)

- Samples are immersed in a solvent (typically water) so avoid samples that degrade in water.