

Preparation Of Powder Samples

A Guide on Preparation of Samples for Delivery to HarwellXPS



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At HarwellXPS, we are happy to receive powdered samples in clean, well labelled and secured **glass vials** which we would mount using standard protocols in our laboratories.

However, some users wish to immobilize their samples prior to shipping and we have prepared this document to aid that process.

Requirements for Mounting

- Silicone-free double-sided tape¹ (less than 10 mm x 10 mm square; 5 mm x 5 mm is ideal)
- Clean metal spatula and aluminium foil
- Clean flat substrate ≤ 1 cm square (*e.g.* stainless steel, silicon wafer or cut glass slide)

Mounting Powders with Adhesive Tape

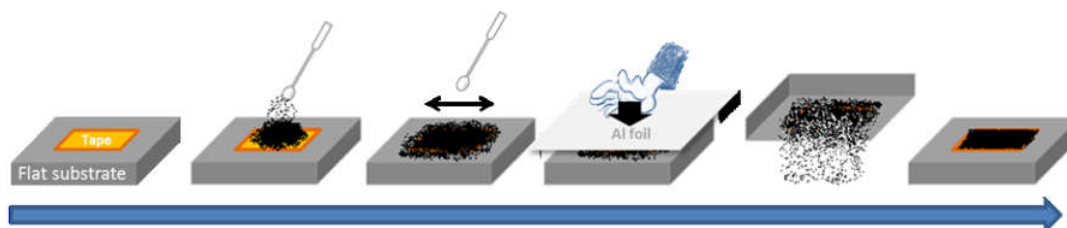


Figure 1. Step-Wise Method for Pre-Mounting samples for shipment to HarwellXPS

Note: In this method of mounting, it is important that no underlying adhesive tape is observed visually so that it will not be observed in the analysis. To achieve this, the tape should be completely covered as possible. If this is not possible, a sample of only the tape should be supplied as reference.

¹ Carbon tape used for SEM analysis is sufficient, but typically contains silicones which may contaminate your surface and affect your analysis. Conducting carbon tape, such as that from Agar or insulating 3M Scotch double sided tape type 665 are recommended.

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Method

As shown in figure 1, the steps are as follows:

1. Place a piece of double-sided tape on a suitable clean flat substrate. Where possible, please keep your tape size to 5 mm x 5 mm. Note that conducting and non-conducting adhesive tape are fine, however we recommend 3M Scotch double-sided tape to minimise possible contamination from polydimethylsiloxane (PDMS).
2. A very small amount of the powder is collected with a spatula and deposited into the centre of the tape.
3. Gently press the powder across the entire surface of the tape. Spread the powder as evenly as possible and uniformly cover the tape
4. Cover the powder with a piece of clean aluminium foil (**with the non-shiny side**), then press the powder to stick on the tape firmly and make a flat surface
5. Invert the substrate to remove the loose powder, and gently tap the side of the sample to remove loose particles. Repeat this until no loose powder comes off the surface. As an extra step to remove the powders, a gentle stream of dry, high purity inert gas (nitrogen or argon) is recommended. **Do not use** any canned compressed air as these will contaminate your surface.
6. Ensuring there is no loose powder on the substrate, wrap your sample in clean aluminium foil individually, with the non-shiny side facing the analytical surface (*i.e.* your powder).