Sample Preparation Policies and Procedures

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1 Available Sample Plates

There are three sample plates available; each plate style has a specific purpose. Figure 1 shows the aforementioned sample plates. Below, we outline their uses:

(a) Standard Copper Sample Plate

- Uses:
  - LN_2 Cooling (down to 80K)
  - LHe Cooling (down to 30K)
  - Sample Heating (up to 450 K)
- Restrictions/Advantages:
  - Limited Sample Area (12 mm X 14 mm)
  - Compatible with RSXS Endstation

(b) In-plane Magnetic Sample Plate

- Uses:
  - XMLD
- Restrictions/Advantages:
  - Limited Sample Area (5 mm X 5 mm)
  - Not compatible with RSXS Endstation

(c) Out-of-plane Magnetic Sample Plate

- Uses:
  - XMCD
- Restrictions/Advantages:
  - Limited Sample Area (10 mm X 10 mm)
  - Not compatible with RSXS Endstation

1.1 Usable Area for Sample Mounting

1.1.1 Standard Sample Plates

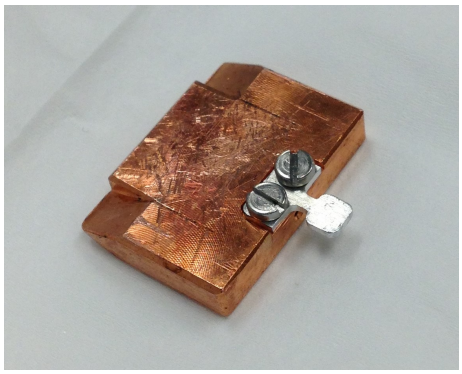
The samples must be mounted within the usable area of the respective sample plates. Failure to comply will result in your material samples falling into the vacuum chambers. Figure 2 shows the usable area for the standard sample plate.

1.1.2 Sample Plates for Magnetic Measurements

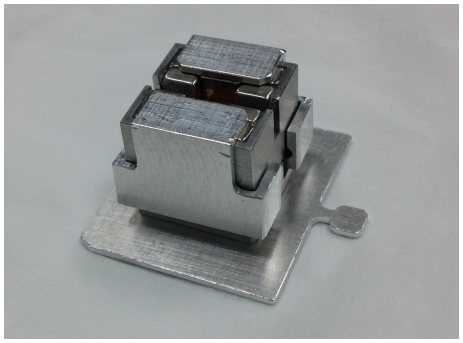
The usable area of sample plates for magnetic measurements have much more restrictions attached.

1. The sample size is restricted to either 5 mm X 5 mm (in-plane) or 10 mm X 10 mm (out-of-plane).
2. Only a single material sample should be affixed to the magnetic sample plates in order to ensure the B-fields are orthogonal or parallel to the material sample surface.
3. There is only one of each — in-plane and out-of-plane — magnetic sample plates available.

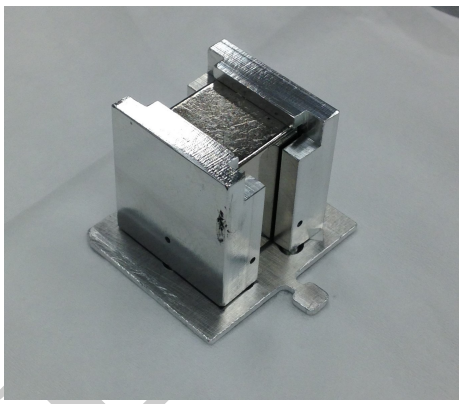
Figure 3 shows the usable area for the magnetic sample plates.



(a) Standard Copper Sample Plate

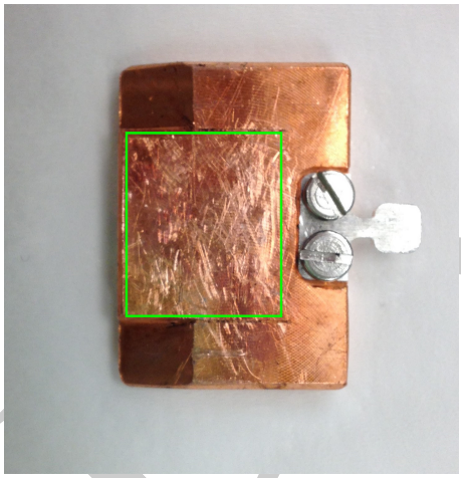


(b) In-plane Magnetic Sample Plate



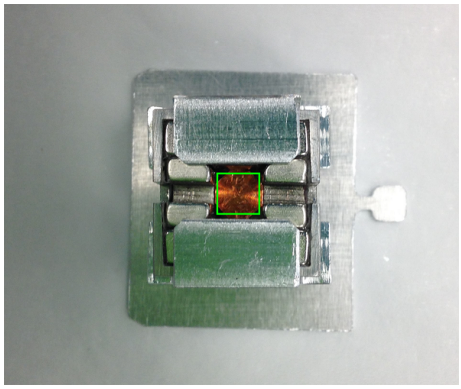
(c) Out-of-plane Magnetic Sample Plate

Figure 1: Available Sample Plates for RIXS/XES Endstation

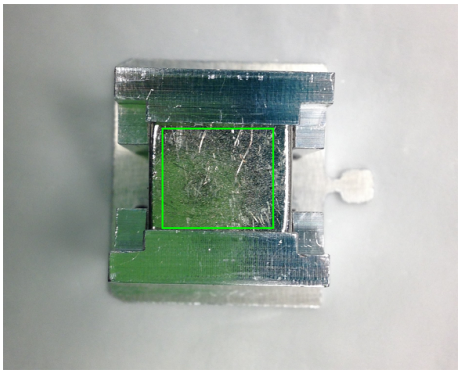


(a) Standard Copper Sample Plate

Figure 2: The usable area for the standard sample plate is indicated by a green box.



(a) In-plane Magnetic Sample Plate



(b) Out-of-plane Magnetic Sample Plate

Figure 3: The usable for the magnetic sample plates is indicated by a green box.

2 Mounting Samples

The most important part of any experiment is the set-up. In this case, this means sample mounting. Below are steps that should be followed to ensure there is minimal cross contamination between your material samples and those other fellow research groups as well as to maintain the general condition of the beamline optics and detectors.

Always ensure proper gloves are being worn during sample preparation.

2.1 Thin Films on Wafers and Crystals

Thin films and single crystals can be mounted quite easily using an adhering conducting tape such are ”carbon tape”. However, there a few steps that should be followed to ensure adequate vacuum in the load-lock chamber and no loss of your material sample.

1. Adhesive tapes tend to significantly out-gas, which can strongly influence the time required for load-lock chamber evacuation. One should always use a minimal amount of adhesive tape; never use more adhesive tape than the area of the material sample you intend to affix. Figure 4 demonstrates this situation.
2. Since adhesive tapes rely on pressure to initiate the temporary bond, always apply firm pressure to the material sample. If the sample is delicate use tweezers near the edge of the sample to apply pressure. Figure 4 illustrates the use of tweezers for applying pressure to material samples.

(a) Use adhesive tape sparingly. (b) Apply firm pressure to sample.

Figure 4: Important aspects of using adhesive tape for mounting materials samples.

2.2 Pressed Pellets and Arbitrary Material Chunks

Pressed pellets and arbitrary materials chunks are mounted in a similar manner to thin films and single crystals, except special care should be taken to ensure that the material sample does not shadow the desired detectors. Figure 5 shows the required sample geometry to avoid shadowing. Additionally, it should be ensured that the material sample is affixed with sufficient contact to the sample tape; this is important when material samples are rough or uneven and failure to do so may result in the sample falling during transfer. To summarize, ensure the following when mounting irregularly shaped material samples:

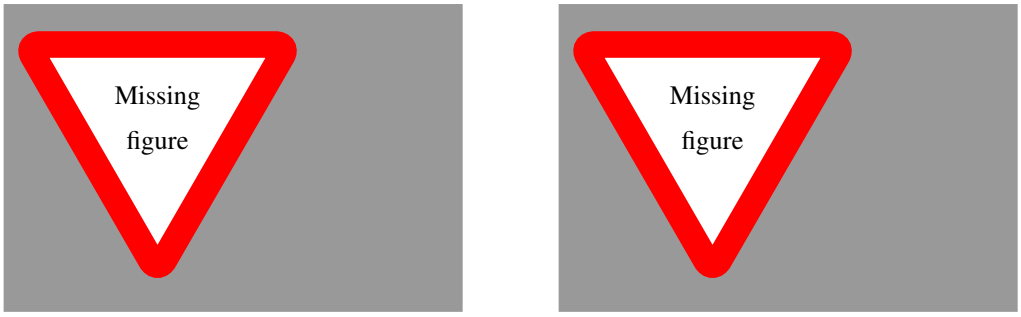
1. The sample surface of interest will face the desired detectors.
2. The surface used to affix the material sample to the sample plate is sufficiently flat.

2.3 Powders

Likely one the most widely used material sample forms, powder material samples provide the majority of the contamination observed in endstation vacuum chambers. Therefore, mounting these material samples requires the most care in order to avoid excessive contamination to the vacuum during the experiment. There are three options for mounting this “sample form”:

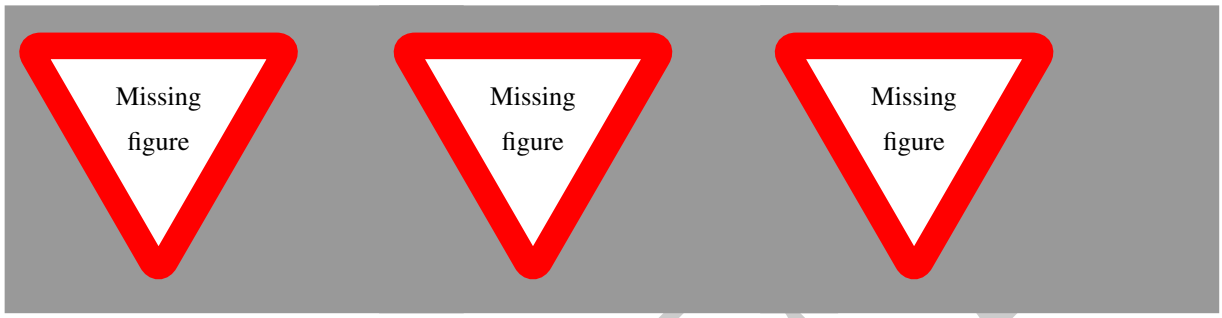
1. Adhered to the sample plate with copper tape.
2. Adhered to the sample plate with silver paint.
3. Pressed into indium foil (or another malleable metal), which is then affixed with carbon tape.

In all cases, indium foil is most desired mounting method. However, if the particulate size of the powder is too small to embed into indium foil, then copper tape oR silver paint should be used, with the later the more desirable.



(a) Good Sample Geometry (b) Bad Sample Geometry

Figure 5: The sample surface should not be shadowed from the desired detectors.



(a) Copper Tape (b) Silver Paint (c) Indium Foil

Figure 6: Mounting a powder material samples.

1. Copper Tape

Pressing powder material samples into copper tape is required if the powder is nano-size. The copper tape is less fibrous than carbon and therefore forms minimal vacant pores during out-gassing. Mounting powder material samples with copper tape is stepped out below.

(1) Cut a small 5 mm X 5 mm (or smaller) squares from copper tape roll.

(2) Remove the powder sample from container using scoop/spatula. A very small amount is required.

(3) Deposit material sample to the centre of the coper tape.

(4) Ensure you close your main container as soon as practically possible.

(5) Work material into the copper tape using a smooth blunt or round tool.

(6) Firmly tap the sample plate on its side to remove loose powder.

- If material is not toxic, use compressed air/gas to remove additional loose particles.
- If the material is toxic, preparation should be done in the wet lab. Make sure you remove any excess powder with a tool.
- Failure to remove excess powder will be visible in the endstation.

(7) Repeat for additional samples.

(8) Ensure adequate separation to reduce contamination. Further reduce contamination by mounting only one sample per plate or in non-similar groups.

(9) Always clean tools after mounting each material sample to reduce contamination.

2. Silver Paint

Silver can provide an easy means to adhere powder material to a sample plate. Please follow the steps below for preparing a powder material sample with silver paint. Please note that 24 hours drying time is required, therefore this preparation technique requires planning. This can be done at your host institution prior to arrival.

(1) Ensure the silver paint is mixed thoroughly and thinned to the appropriate viscosity. (follow manufacturings directions)

(2) Do not apply silver paint directly to the sample plate. Cut small 5 mm X 5 mm (or smaller) squares from a rigid foil, such as copper or iron.

(3) Affix the metal squares to the sample plate with carbon tape. Never user more carbon tape than the size of the square. These can affixed to a dummy sample plate at your host institution and transferred to the beamline sample plates at a later time.

(4) Apply silver paint to metal square, keep paint to the centre and ensure even coverage.

(5) Remove the powder sample from container using scoop/spatula. A very small amount is required.

(6) Deposit material sample, sparingly, but still ensuring full coverage.

(7) Ensure you close your main container as soon as practically possible.

(8) Allow paint to dry tack-free for 30-60 mins, depending on conditions.

(9) Firmly tap the sample plate on its side to remove loose powder.

- If material is not toxic, use compressed air/gas to remove additional loose particles.
- If the material is toxic, preparation should be done in the wet lab. Make sure you remove any excess powder with a tool.
- Failure to remove excess powder will be visible in the endstation.

(10) Repeat for additional samples.

(11) Ensure adequate separation to reduce contamination. Further reduce contamination by mounting only one sample per plate or in non-similar groups.

(12) Allow sample plate to cure for 24 hours.

(13) Always clean tools after mounting each material sample to reduce contamination.

3. Indium Foil

Pressing powder material samples into indium foil solves several problems associated with using fibrous adhesive tapes. The foil is, to a large extent, vacuum compatible, and does not out-gas in vacuum causing the release of material into the vacuum chamber. It also does not form vacant pores during out-gassing, which can result in large contributions from adhesive tapes in the experimental data. Mounting powder material samples with indium foil is stepped out below.

(1) Cut small 5 mm X 5 mm (or smaller) squares from indium foil or equivalent malleable metal.

(2) Affix the metal squares to the sample plate with carbon tape. Never user more carbon tape than the size of the square.

(3) Remove the powder sample from container using scoop/spatula. A very small amount is required.

(4) Deposit material sample to the centre of the foil square.

(5) Ensure you close your main container as soon as practically possible.

(6) Work material into the foil using a smooth blunt or round tool.

(7) Firmly tap the sample plate on its side to remove loose powder.

- If material is not toxic, use compressed air/gas to remove additional loose particles.
- If the material is toxic, preparation should be done in the wet lab. Make sure you remove any excess powder with a tool.
- Failure to remove excess powder will be visible in the endstation.

(8) Repeat for additional samples.

(9) Ensure adequate separation to reduce contamination. Further reduce contamination by mounting only one sample per plate or in non-similar groups.

(10) Always clean tools after mounting each material sample to reduce contamination.