EDXRF User Training

5. Sample Considerations
Sample Loading

Quick and easy specimen presentation

primary capillary

adhesive tape
(sticky surface up)

detector

“acrylic” X-Y-Z sample-stage
Sample Loading

An alternative sample presentation

(minimizing bulk of specimen support)

primary capillary

adhesive tape
(sticky surface up)

detector

X-Y-Z sample-stage

X

Y

Z
Scatter Reduction From Sample Loading

Reduction of “overspill-scatter” with method of specimen presentation

~200×100µm Fe-base particle spectra (when illuminated by a 300µm beam)
Primary Beam Overspill (300µm)

- ~150×200
- ~100×50
- ~300×300

overspill
Scatter of Primary Beam Overspill

300µm monocapillary; Fe-alloy particles on sticky tape directly on acrylic sample stage.
Scatter of Primary Beam Overspill (2)

FeKα & β sum peaks

MoKα

“bulk”

MoKβ

RhKα & Kβ Compton scatter “artefact” peaks

RhKb

“particulate” on sticky tape

10 15 20 25 keV
Glass Particle Presentation

100×50µm particle directly on stage - a worst case scenario

1000×600µm particle on 6µm Mylar located 5cm above stage

Si
Ca
Fe

RhKα Compton
RhKβ Compton

300µm capillary Rh-anode tube
Glass Particle Presentation

- 6µm Mylar
- adhesive
- glass particle

- primary capillary
- detector

- X-Y-Z stage
- 8x12

- 6µm Mylar
- adhesive
- glass particle

- X-Y-Z stage
- 8x12
Comparing Thick and Thin Samples

specimens of a steel alloy in 4 to 8 keV range - relative analyte intensities equal
specimens of a steel alloy in 16 to 20 keV range - relative analyte intensities NOT equal
Effect of Sample Thickness on Intensity

- Sample surface
- 50% (of critical depth)
- Critical depth

90% (of maximum possible intensity)

% of maximum available intensity

% of “critical depth”
Summary of Handling Various Sample Types

- Fragments, chips, particles, loose powders
  - “Carbon tape bridge”:
  - Mylar “sandwich”:

Adhere the material onto the adhesive side of the tape, feel off the back layer, and “bridge” it across an elevated sample cup.

Pour some of the material on a layer of Mylar supported by a double open-ended sample cup. Then place another layer of Mylar over that, and clamp it down with the O-ring.

**Benefit → Safe use of vacuum!**
Summary of Handling Various Sample Types

• Large, irregular shaped samples:
  – *Fun-tack, or Putty can be used to adhere an irregularly shaped sample to the stage*
    • Putty should not be in the way of the X-ray, to avoid signal interference

• Fabric (i.e. gunshot residue cloths)
  – Sample cup shown in example 1 can be used to stretch a fabric

• Liquids, resins, etc. → NO VACUUM!
  – Either held in an uncovered cup (not recommended), or a special liquid sample cup that can be covered and bubbles eliminated:

  Conical shape allows:
  a) bubbles to be eliminated from the analysis area, and b) can be covered with Mylar to prevent spillage
Summary of Handling Various Sample Types

- High volume samples – arrays, sample holders, trays
  - If your laboratory has sample array holders, they can be fitted directly onto the Orbis stage.
  - The software allows quick and easy recollection of the array coordinates, regardless of whether or not the sample tray is loaded in at the same orientation/position.