OTJA – HYSPEC Sample Mounting Aid

Mounting and aligning your sample

1. Materials
    a. Good
        i. 'Transparent' like Aluminum, alumina or single crystal silicon
            1. Still introduces background, so less / thinner is better
            2. Available at HFIR and SNS
                a. Aluminum 6061 thin plates
                b. Aluminum posts and clamps
            3. Recommend any fasteners in beam elevation be Al, or even better, moved away from beam elevation
            4. Wrap your sample with foil to add marks for alignment ID
            5. Aluminum wire to secure sample
        ii. 'Absorbing' to shield against material just above or just below the sample
            1. Cadmium sheet
            2. Gadolinium Oxide spray
            3. Boron nitride spray
            4. Boron nitride solid (careful, it's fragile. On the bright side it's also an electrical insulator)
            5. Boron carbide sintered material
            6. All available at SNS
        iii. Thermal conduction
            1. Indium for $4\ K < T < 300\ K$, but keep it away from the beam
    b. OK but not great
        i. Teflon tape: no hydrogen, and high temperature compatible up to a point
        ii. Vanadium: may be needed for high temperature, and better for pure diffraction, but is a stronger scatterer than aluminum overall so more inelastic background for DGS instruments
    c. Bad
        i. Not terrible if you can shield against it above or below the beam…
        ii. Scatters like crazy
            1. Copper
            2. Stainless steel
                a. Please note that you can and should purchase aluminum fasteners and washers!
            3. Most glues (Hydrogen is a strong isotropic scatterer)
            4. Note that any scatterer like this off-center from the sample scatter will provide significant angle dependent background
        iii. Activates like crazy

1. Sometimes your sample itself will activate. To check that we do have a sample activation tool.
2. Stainless steel (cobalt inside)
3. Indium (keep away from beam elevation)

iv. Doesn't like higher temperature
1. Cadmium or indium, > 300 K
2. Aluminum, > 700 K

v. Best for sub-1.5 K
1. Copper doesn't go superconducting like aluminum. Superconducting aluminum is a lousy thermal conductor. Aluminum fasteners may be used so long as they do not directly contact the sample, or if one can cool in field to stop the transition to the superconducting state.

vi. Strong magnetic fields
1. Stainless steel is too magnetic
2. Go instead with aluminum, Brass and OFHC copper.

2. Attaching the sample to your mount
   a. Wrap with aluminum foil
   b. Aluminum wires
   c. Acceptable glues
   d. Why it needs to be secure
      i. Some sample environments hammer your sample
      ii. Temperature changes (especially high temperatures) cause expansion issues (which is why I'm not so thrilled with the screw-in-to-secure strategy in general)
3. Integration with sample environment
   a. Sample volume
      i. More extreme sample environments generally provide smaller volumes for the sample, for pressure and low temperature
   b. Attachment geometry
      i. Generally need to connect with a tapped hole using a tapped hole (we have studs)
      ii. Know attachment point to nominal beam elevation. HYSPEC doesn't have a sample elevator…
   c. Temperature confidence
      i. Tapped 4-40 hole close to sample can help, but need wire to have access
      ii. He gas via convection/conduction
         1. Helium can
            a. Used to ensure temperature stability
            b. Usually back filled at atmospheric pressure

            c. For weakly scattering samples and low temperatures, runs the risk of He recoil
            d. For temperatures below 2.6 K, runs the risk of liquid He roton scattering
        2. Top loading, low pressure Helium exchange gas (less He recoil)
    d. Material limitations
        i. High temperature: Cd bad for example
4. Compatibility with Instrument
    a. Size of the sample needs to be compatible with the size of the neutron beam cross section
    b. Strong attenuators need to be smaller or thinner
    c. Time-of-Flight related spurious signals mitigation
        i. Minimize the material surrounding the beam. A BNL or HFIR standard single-crystal can introduce a flat (dispersionless) energy loss feature, due to the possible scatter from the sample then backscatter from the can.
        ii. Shield up and down. A similar flat (dispersionless) energy loss feature can appear from scatter directly up or down, then out to the detector.
5. Alignment concerns
    a. Alignment opportunities
        i. Neutrons: really best for thick samples
            1. CG1
            2. Separate neutron scattering facility
        ii. X-ray Laue: sensitive to surface, but maybe not the bulk
    b. Single crystal guidance
        i. Aluminum disk vs rectangular sheet: disk provides extra rotation axis
        ii. Available mounts
            1. Show the posts with rotation, angles, etc.
            2. Goniometer style
    c. Multiple crystal guidance
        i. Stacked plates, minimizing aluminum material in beam
        ii. Changing orientations convenient – pre-oriented and marked plates.
    d. Powder guidance
        i. Cylinder containers
        ii. Annular containers
        iii. Flat cells
6. Helpful anecdotes
    a. My sample dropped off!
    b. My sample mount unscrewed!
    c. Why did my sample bend out of beam?
    d. This looks like a resonance, but not quite…