Powder Camera Sample Preparation --- 2/2/2011

The Debye-Sherrer x-ray camera (powder camera) requires that a cylindrically shaped sample be placed and rotated at the center of the cylindrical camera. As the x-ray beam is less than 2 mm wide as the x-rays impinge upon the sample, the sample need only be 3 mm in any dimension. The student is required to fabricate such a sample which will fit onto the camera's axial spindle via the sample holder (1/8" diameter slip fit).

Sample preparation is critical for producing the high-quality x-ray films that can be used to determine the crystal structure and the lattice parameters. There are several techniques to fabricate such a sample of which the following as just a few. Which technique works the best depends on the material, x-ray energy, and operator skill. However, as the x-ray energies are fixed, and the samples are known, operator skill during sample preparation has a significant impact on the quality of the results.

Figure 1 shows detailed views of the powder camera sample area. (Removing the disc-shaped camera lid will produce the same view.) The sample diagrammed in red, needs to be of the appropriate size and appropriately attached to the sample holder. The sample spindle can be rotated by spinning the pulley on the camera back. The sample is a polycrystalline power, which must be contained somehow and attached to the sample holder. In the figure 1 illustration, the powder (sample, shown in red) is contained in a thin sphere (black border around the red sample), and attached to the sample holder with chewing gum (blue-green) or other appropriate adhesive. Before information about the sample structure (and the physics therein), other design parameters need to be determined (and physical insight is necessary here as well).

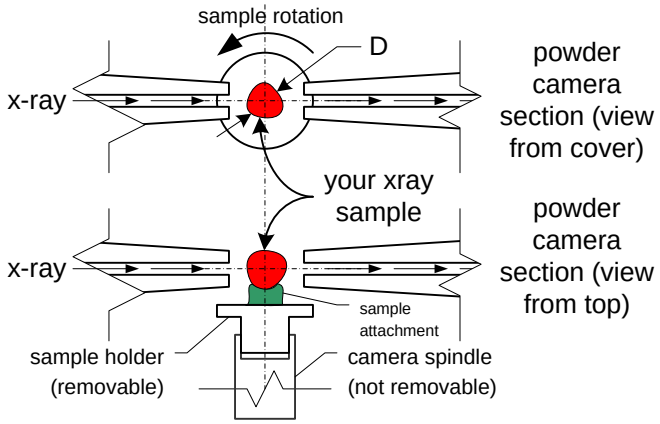

Figure 1, Interior Detail of an x-ray Powder Camera, Sample Area

The first design parameter of interest is D, the diameter of the sample through which the x-rays pass, diffract, and are absorbed. If D is too small, most of the x-rays go around the sample resulting in very long exposures. If D is too large, the forward x-ray diffraction will be absorbed in sample. It should be clear then that a polycrystalline Pb sample can have a much smaller D than a polycrystalline Al sample.

It should also be clear that the x-rays pass through whatever is holding the powder together (a balloon in this hypothetical case) and this whatever could also diffract x-rays and will absorb x-rays. Note that the air in the camera absorbs x-rays (how much?). It should be obvious that a Pb balloon would be a poor choice to contain the powder, whereas a thin plastic or rubber balloon might be an acceptable choice.

Figure 2 shows various sample preparation methods (**contain** and **attach**) for the powder camera. In practice, (practical side of physics) the sample needs to something that can be prepared using gloves (good safety practice) by someone (aspiring physicist) with reasonable dexterity (sans coffee) in a less than an hour and at reasonable cost. Reasonable cost is usually synonymous with either cheap materials (consumables, as in lab fees) or re-useable material (like the sample holder).

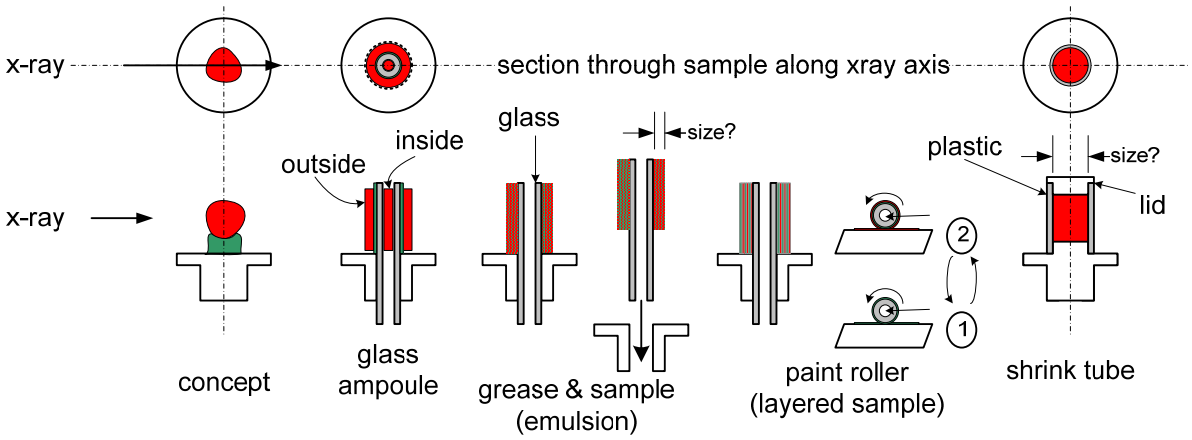

Figure 2, Some Methods to **Attach** the Xray **Sample** to the Sample Holder

The conceptual sample shown in figure 2 is as in figure 1. The glass ampoule sample holder uses a thin glass tube that slides into a hole in the sample holder. If the powder can be packed into the ampoule and contained on the ends, then glass ampoule provides an elegant solution for sample preparation. (What is the effect of using quartz ampoules in place of glass ampoules?)

In practice, as D (inside diameter of the ampoule) is fixed there isn't any way to add more sample to the x-ray path unless the outside of the ampoule is coated with the sample powder. Such a coating would require an adhesive, which the x-rays would pass through. The adhesive or binder decreases the effective sample density, absorbs x-rays itself, and could produce an x-ray diffraction signal of its own. Needless to say, the percentage of binders in a sample needs to be kept to a minimum.

One technique to coat the sample onto the ampoule is to first coat the ampoule with a non-water soluble grease (silicone high vacuum grease is one choice), then roll the greased ampoule over a thin layer of the sample. Invariably, in order to build up a sufficiently thick sample coating, the grease mixes with the sample, forming a paste. (Note: The ampoules themselves, being tubes can be conveniently handled by stripping a piece of the solid BSC #28 wire, exposing about 2 cm of wire. The tinned copper will fit inside the tube, the insulation will not. The rest is self explanatory.) The advantage of the paste is that it can be coated on the ampoule until a satisfactory preparation is achieved. The disadvantage of

the grease is that it is soft and is easily damaged. Recall that photographic film is loaded in the camera in complete darkness and that the sample is exposed and only 5 cm from the film surface. (It's prudent to practice loading the camera with the dummy films in the dark room before wasting several hours of expose time.)

The ampoule may also be coated by layering the sample with alternating coats of adhesive. Once an ampoule has been roughened with 500 grit SiC paper, it can be attached to a wire and the combination bent to resemble a paint roller. (A paint roller is an implementation of a wheel & axle, from basic mechanics.) The roller (ampoule) is coated with adhesive (clear fingernail polish thinned by 10% in acetone) by first adding a drop of adhesive to a flat, non-absorbing surface, then passing the roller over the drop, flattening the drop, and coating the roller with adhesive. Before the adhesive dries (within 10 seconds) the roller is rolled across a thin layer of the sample, which adheres to the adhesive. Using a heat gun on low and at 5 cm from the output, rotate the sample for about 30 s to dry the adhesive. Once cooled, the sample should be attached well enough to the ampoule that it is not easily removed. Figure 2 shows a paint-roller (layered) coated sample.

The above description of alternate coating can be repeated to increase the sample size, D, that is add layers of sample. Before additional adhesive is applied, the previous sample/adhesive layers must dry. Samples can be reworked by removing the adhesive in acetone or fingernail polish remover, or simply starting with a new ampoule. It is recommended that both lab partners prepare a sample and the best one exposed to x-rays.

There is a practical size limit to the paint roller technique. Above 0.5 mm coating, the sample surface becomes rough and 1 mm is about the limit for a sample/adhesive sanwich. For samples with D over 2 mm, the sample holder is modified so that a hollow plastic tube can be attached. These tubes are heat shrinkable, that is, they contract and harden on exposure to about 200 C. There are literally hundreds of different types of heat-shrinkable tubing. This clear tubing is probably Kynar (for x-ray absorption use any convenient hydrocarbon) with a 0.2 mm wall thickness. The opaque tubing is probably polyolefin.

Using a razor blade, cut a 12 mm length of tubing, square on both ends, and slip it over the sample holder. Place the sample holder in a 1/8" hole in a flat plate so that the open tube end is up. Using a creased piece of paper or foil as a guide, fill the tube with the sample. Tap the side of the tube until about 2 mm of tube is unfilled, then slip the cap over the tube. While leaving the sample in the 1/8" plate, heat all the way around the tubing for 20 seconds (heat gun on low at 5 cm distance). If the sample is not axially aligned to the sample holder, it may be moved slightly with tool pressure before it cools. If the sample preparation is unacceptable, use the razor blade to separate the tubing from the sample holder and lid and try again. The sample holders and lids are small, so that considerable manual dexterity is required to avoid hurling them across the lab.