In the next couple of classes we will learn about x-ray diffraction. This is an amazing technique with long history of applications to: **Chemistry and Biology**, structure solving of crystals from NaCl to insulin to hemoglobin to DNA and even viruses!; **Geology**, identification of minerals even mixtures such as clays; **Material Science:** identification of alloys and phases, assessment of crystalline quality, identification of stress, and grain size. For NanoTechnology the Material Science topics are the most important. It is key to understand that this is a measurement of the average characteristics of a population or an ensemble average.

In the first class we will quickly review x-ray diffraction.

This will include:

- An Introduction,
- X-ray generation and naming x-ray lines;
- A review of crystals
- Diffraction leading to Bragg's Law (very constraining)
- Diffraction of real samples: crystal size, strain
- Diffractometry our Bragg-Brentano Instrument
- Phase Identification
- Polycrystalline Aggregates Scherrer Formula for Size determination

At the same time!, as a group, we will also prepare and run one powder sample of some TiO_2 nanoparticles.

On the web you will find four excel files with Θ -2 Θ diffraction data (patterns).

http://www.nhn.ou.edu/~bumm/NanoLab/downloads/X-ray_Data/

These have an assortment of materials including glass, gold NPs on glass, flat gold NPs, TiO_2 nanopowders and sunscreens.

I will show how we want to plot these. Then over the next week we want to do the following:

- 1. Identify all of the Bragg peaks in your data that are not due to the substrate or other artifacts.
- 2. From those peaks, assign each of them to a Bragg reflection (its Miller index). You may not be able to observe all of the peaks that are in the ICCD. This is to be expected. Some are quite weak. The purpose of the ICCD is to guide you a) where to look for the peak, and b) assign the Miller indices for the peaks.
- 3. For the TiO_2 sample you may have two components, anatase and rutile. Each of these needs to be analyzed in a separate tables (for Bragg and for Scherrer). So for the TiO_2 sample you will have some peaks for Anatase, some for Rutile.
- 4. Enter Bragg angles into you table with their Miller indices.
- 5. Calculate the inter-plane spacing for each Bragg peak.
- a) For Au, calculate the lattice constant for each brag reflection and average them to give your result for the lattice constant.
- b) For ZnO and TiO2, calculate the appropriate lattice constant only for those peaks I flagged in the table. You will get two sets of lattice constants. Average each set separately.

- 6. Measure the peak widths and enter them into the Scherrer table.
- 7. Calculate the thickness for each peak. The particles may not measure the same thickness in each direction.
- 8. Texture, compare the relative intensities of the Bragg peaks in your sample to the relative intensities in the ICCD. Give a qualitative statement about the texture of the sample. (Which Miller index is parallel to the substrate?) Not all samples will have a texture that deviates for the random oriented texture of the ICCD.