X-Ray Powder Diffraction of Al_2O_3

Experiment #3

Characterization of Materials (96.445/545)

Meg Noah

Objective

The purpose of this lab is to find out if we can determine the grain size based on the X-Ray Powder Diffraction data.

Experimental Equipment

The experimental equipment used for this lab included:

- Powdered metal oxide samples Number 3 and Number 4
- The inXitu BTX system of hardware and software
- The XPowder software on the laboratory computer

The BTX system and XPowder software were described in detail in the last lab report.

Procedure

1. **Sample Preparation**
   The samples were already prepared by the instructor, and labeled with a number although we are told that they are both Al2O3. We are not told the crystalline sizes, but simply that they are different. To be compatible with the BTX, the sample has to be dry, small enough to pass through a 150 micrometer sieve, and large enough to not stick together and to convect inside the sample holder. At least 15 micrograms are needed in the sample holder.
2. **Loading the BTX specimen holder**
   We turned on the BTX, removed the sample holder (already cleaned), and began to load the samples into the specimen holder using the shake option. This step took the longest amount of time, and at the end of the laboratory, we were unable to load the specimen holder with the equipment provided and following the instructions provided to acquire data with sufficient signal-to-noise for post-processing. The main problem that we had was the powder kept caking into into little balls and getting stuck at the top of the vile on the BTX specimen holder. This was probably due to a) humidity, b) clumping already existing in the provided specimen, and c) the ultrasound shake contributing more to caking than to loading.
3. **Acquire exposures.**
   First we tried Number 8, but didn't get quality data. So then we did Number 9. After acquiring data for Number 9, we tried Number 8 again. In both cases, the XRF data identified Ti, so we concluded that the compounds were both TiO_2.
4. **Save Exposures and Data**
   We saved the data to disk, and loaded it into XPowder to find the peak intensities, positions and FWHM.

Results and Discussion

Elements identified with X-Ray fluorescence

The compounds were both Al2O3 and can't be chemically determined by X-Ray fluorescence.

Structure found in XPowder

Figure 1: Sample 3 Unit Cell Peak Lines and Intensities

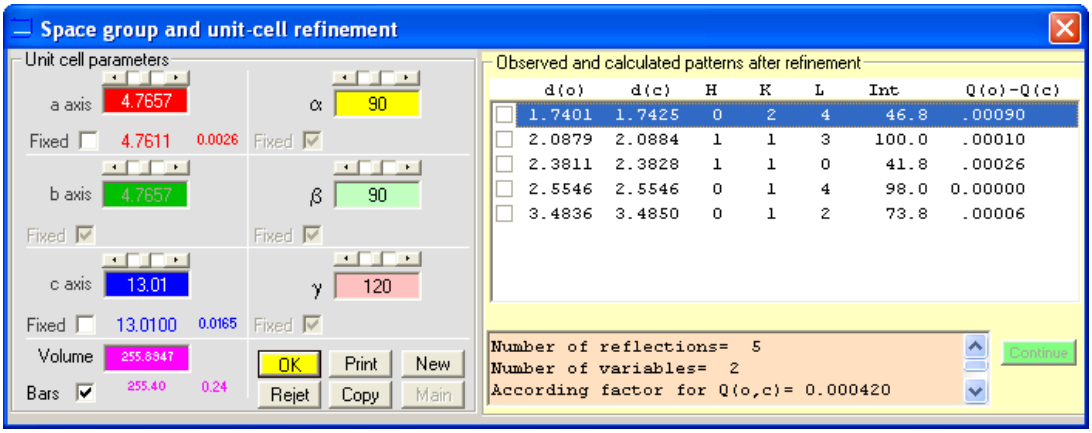
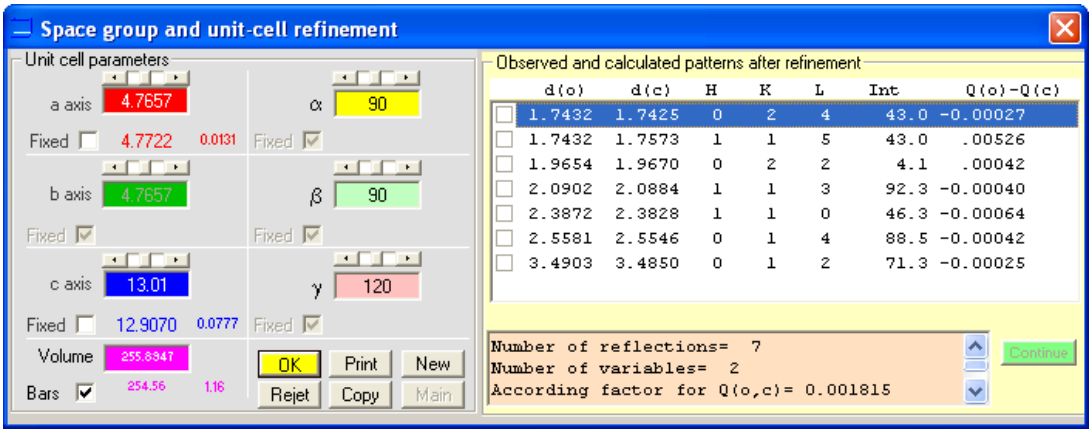
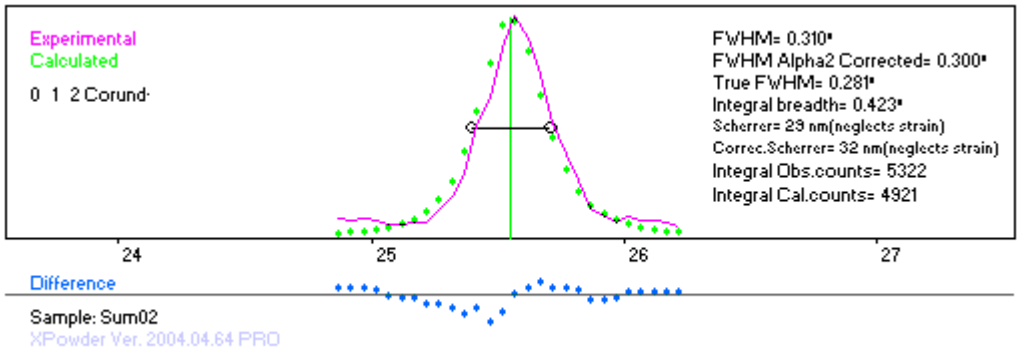
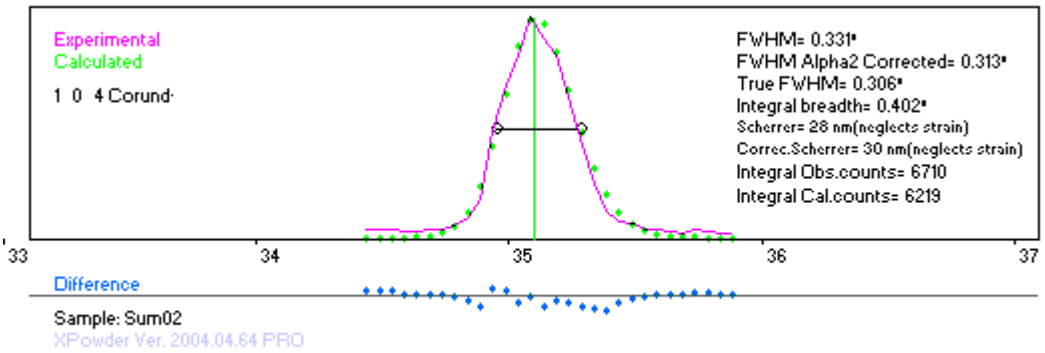


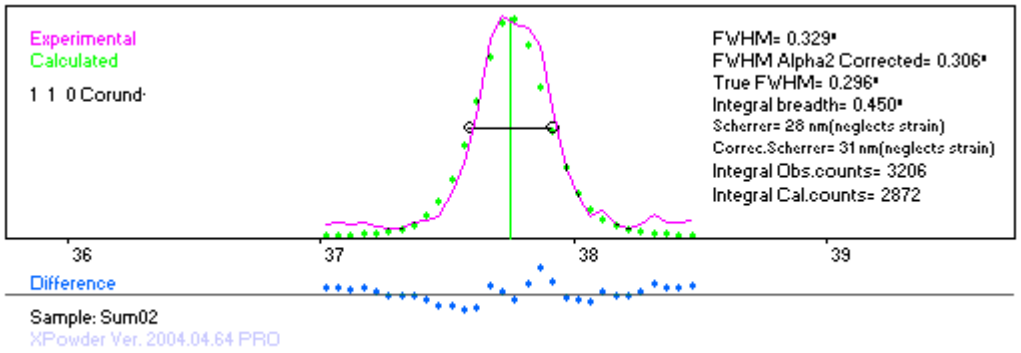
Figure 2: Sample 4 Unit Cell Peak Lines and Intensities

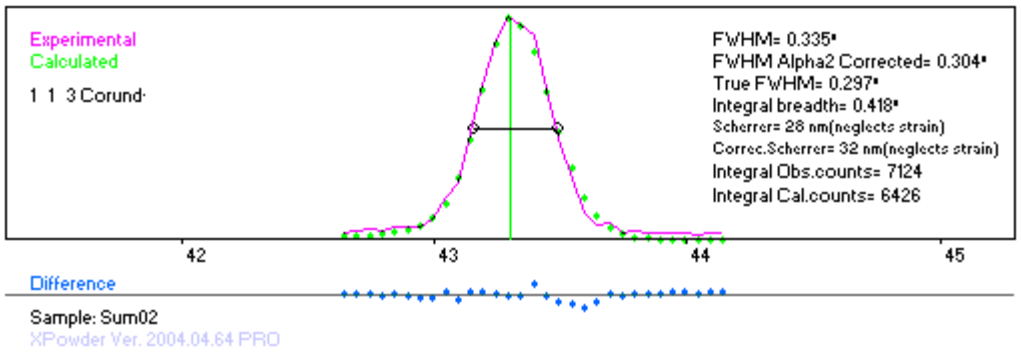


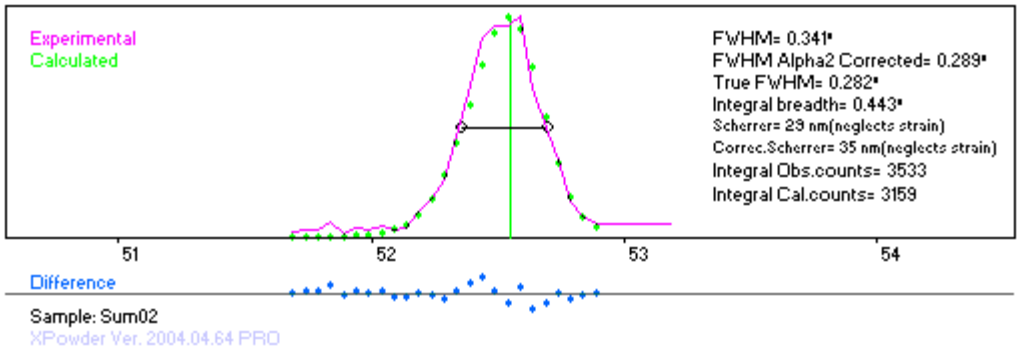
Sample 3 Line Plots



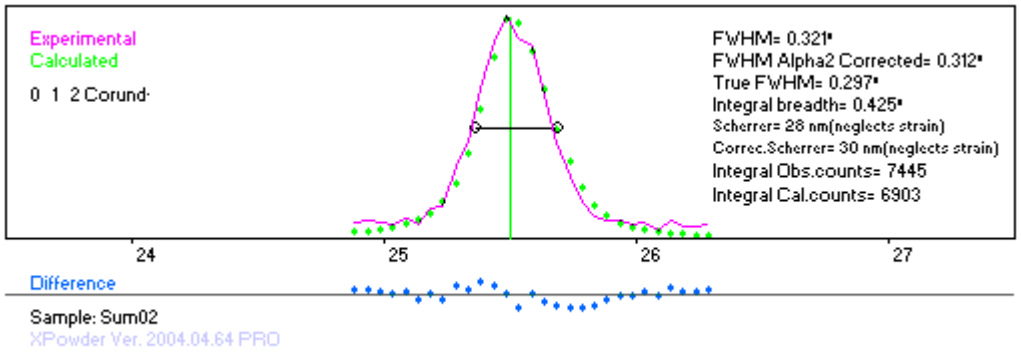


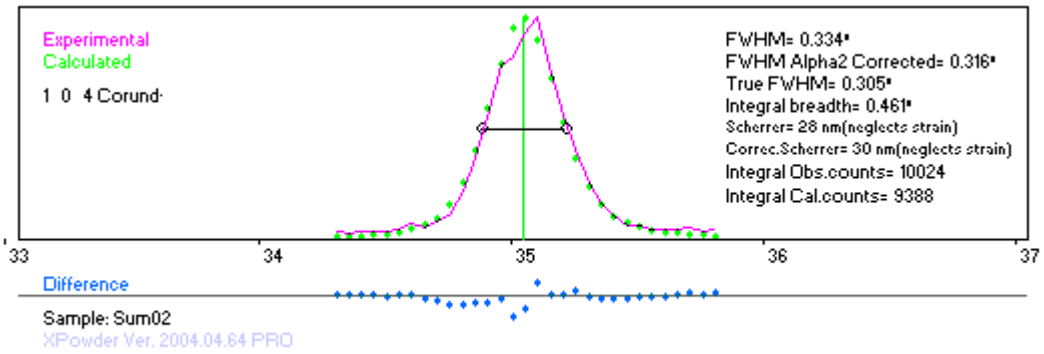


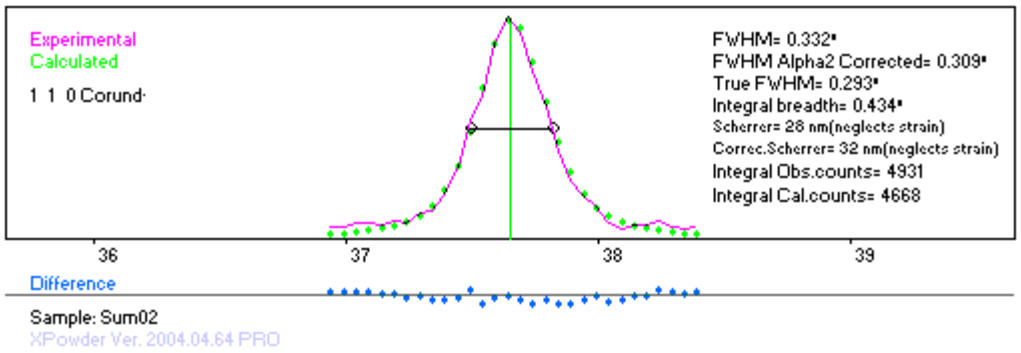


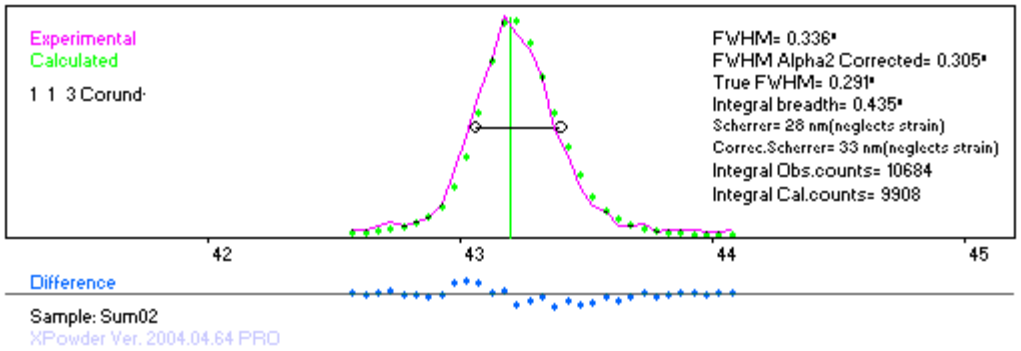


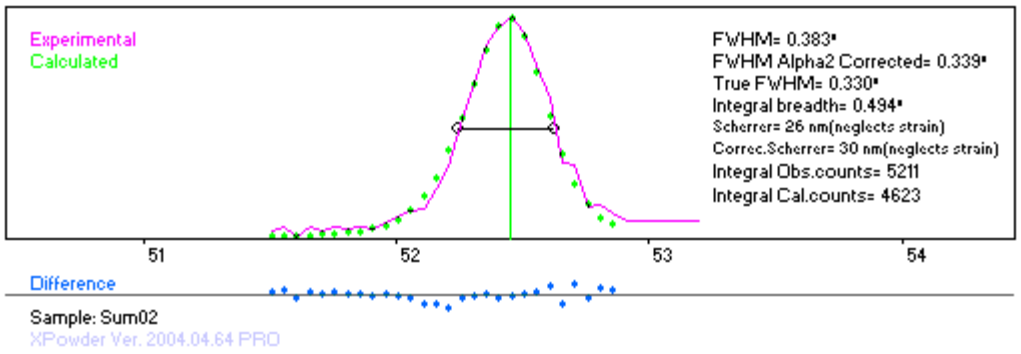
Sample 4 Line Plots











Conclusions

Table 3 shows the measured corrected scherrer sizes for four different X-Ray lines.

Table 3: Corrected Scherrer Sizes for Sample 3 and Sample 4 Lines

h	K	l	Sample 3 Intensity	Sample4 Intensity	Sample 3 Corrected Scherrer Size	Sample 4 Corrected Scherrer Size
1	1	3	100.0	92.3	32 nm	28 nm
1	1	0	41.8	46.3	31 nm	32 nm
0	1	4	98.0	88.5	30 nm	30 nm
0	1	2	73.8	71.3	32 nm	30 nm

The Scherrer equation can be used to determine the crystallite size. It uses a shape factor in x-ray diffraction and crystallography to correlate the size of sub-micrometre particles, or crystallites, in a solid to the broadening of a peak in a diffraction pattern. In the XPowder software, this equation is [1,2,3,4]:

$$\text{Size}(\mu m) = K \cdot \lambda(Å)/(10 \cdot \beta \cos\theta)$$

where K is the shape factor, λ is the x-ray wavelength, β is the line broadening at half the maximum intensity (FWHM) in radians, and θ is the Bragg angle. Values for the shape factor, K (0.8>K>1.1) are experimental constants, which is different when FWHM or β are used. The result is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size.

Since the Scherrer equation is limited to nanoparticles, and not applicable to grain sizes greater than 0.1 µm. In this lab, we've shown that we can't measure the particle sizes that are compatible with the X-Ray Powder machine, and that we can't determine which sample had a larger grain size. What we did determine was the domain size within the crystal, and we showed that this is independent of the grain size of the particles.

References

[1] Martin, J.D. (2008) XPowder, A Software Package for Powder X-Ray Diffraction Analysis, User Guide, Version 2004.04.50 http://www.xpowder.com/download/xpowder.pdf

[2] http://en.wikipedia.org/wiki/Shape_factor_(X-ray_diffraction)

[3] P. Scherrer, Göttinger Nachrichten Gesell., Vol. 2, 1918, p 98.

[4] Patterson, A. L., The Scherrer Formula for X-Ray Particle Size Determination, Phys. Rev. 56 (10): 978–982. 1939.