



### Phase Identification and Crystallite Size Measurement Using X-ray Diffraction

X-ray diffraction (XRD) is a non-destructive characterization technique which can be used for the identification and structural characterization of single crystal and polycrystalline materials. A surprisingly large number of materials fall into this category – metals, ceramics, salts, some polymers, semiconductors, and even table sugar. Unknown compounds can be identified by comparing a sample's diffraction pattern to a library of patterns measured from over 400,000 materials. The peak positions, widths, shapes, and relative intensities of an x-ray diffraction pattern all give additional information about the solid-state structure and composition. X-ray diffraction is a powerful tool for many industries and applications, such as identifying mineral compositions of geological samples, measuring phase purity for quality control, or distinguishing between phases with identical chemical compositions such as austenite and ferrite in steel.

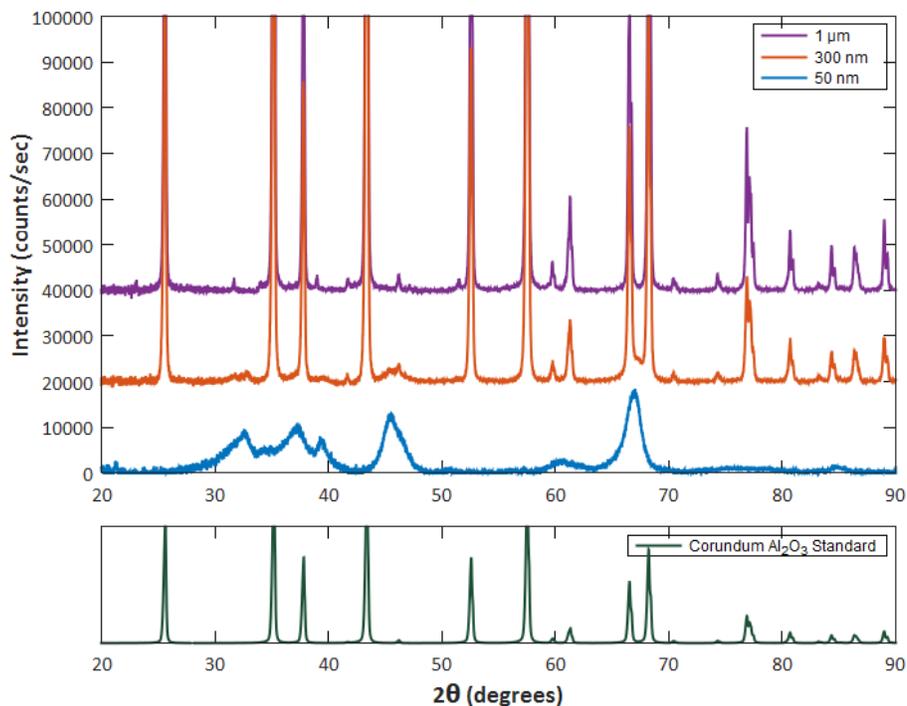


Figure 1. X-ray diffraction patterns of 1μm, 300 nm, and 50 nm alumina powders (top) and x-ray diffraction pattern of corundum from database of standard diffraction patterns (bottom).

The XRD patterns of alumina polishing powders with nominal particle sizes of 1μm, 300 nm, and 50 nm were measured using Rigaku SmartLab X-Ray Diffractometer. Figure 1 shows the experimental results from the three samples in comparison with the reference pattern for corundum from a diffraction file database. It can be seen that the peak positions and intensities of the 1μm and 300 nm particles match those of corundum, the same form of Al<sub>2</sub>O<sub>3</sub> as in ruby and sapphire.



The 50 nm particles have distinctly different diffraction pattern from the 300 nm and 1µm particle samples. The increased peak width is a result of the reduction in crystallite size. The missing and shifted peaks indicate that the structure has been significantly altered from perfect corundum. While chemically it may still be alumina, the structure might have more imperfections, damages or plastic deformation. Plastic deformation in crystals is accommodated by cracks, voids, and strain, all of which produce peak shifts and broadening as seen in the 50 nm sample.

In addition to peak positions and intensities, the width of the diffraction peaks also yields information about the sample’s microstructure. The crystallite size and microstrain have different dependencies on the x-ray scattering angle. Figure 2 shows a Halder-Wagner plot used to independently measure the strain and crystallite size of the sample.

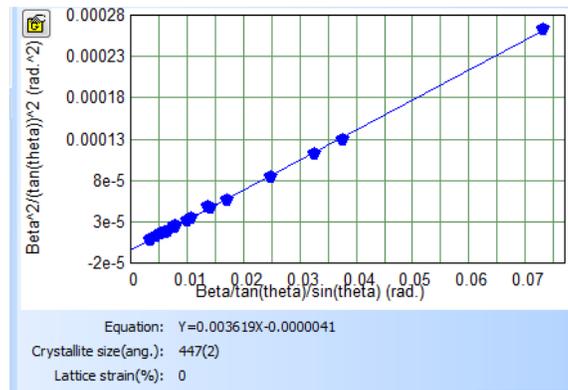


Figure 2. Halder-Wagner plot for the XRD pattern from 1 µm sized alumina particles.

Table 1 shows the results of the size and strain analysis for the three samples. Grain size plays a role in nearly every important material property, such as hardness, ductility, diffusion rates, resistance to corrosion, electronic conduction, and magnetic properties. The high density of grain boundaries in the 50 nm particles is likely to make them harder (requires more force to induce plastic deformation) than the particles with larger crystallite size. Since it takes more energy to move a dislocation across a grain boundary than through the middle of a grain, decreasing the grain size generally increases the hardness of materials.

Table 1 Average Crystallite Size and Lattice Strain of Polishing Alumina Powders

Particle Size	Crystallite Size (nm)	Strain
50 nm	2.14 ± 1.8	0%
300 nm	33.5 ± 0.6	0%
1 µm	44.7 ± 0.2	0%