X-Ray Diffraction and Scattering Equipment and General Area Detector Diffraction System (GADDS)

The materials characterization laboratory is equipped with state-of-the-art analytical x-ray diffraction instrumentation and software. The facility has two independent diffractometers (Figure 1) enabling the study of both polycrystalline and single-crystal specimens. A fully automated Bruker D5005 vertical goniometer system is available for high-precision crystallographic investigations of powder or ceramic samples. Applications for this instrument include qualitative and quantitative phase analysis, crystallite size (Figure 2) and lattice strain measurements, precise lattice parameter determinations, profile shape analysis, and structure refinement by the Rietveld method.

Figure 1: State-of-the-art analytical x-ray diffraction instrumentation. The diffractometer on the right-hand-side makes use of a General Area Detector Diffraction System (GADDS).

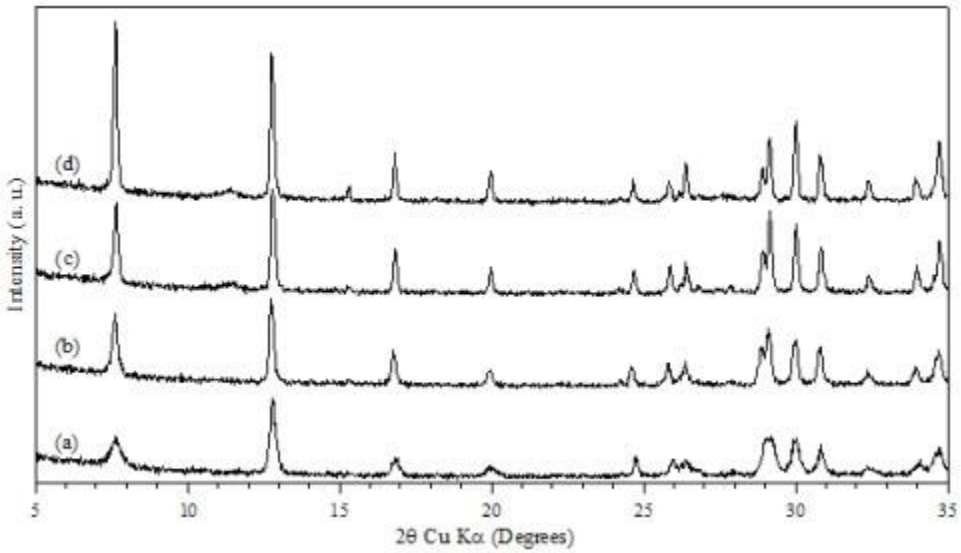

Figure 2: XRD patterns of the ETS-4 products grown at 448 K from synthesis mixtures with molar composition: 3.6SiO2 : 1TiO2 : 5.5Na2O : xH2SO4 : 230.2H2O; where (a) x=4.4, (b) x=3.6, (c) x=3.4, and (d) x=3.3. The broadening of the XRD peaks of products grown at x=4.4 and x=3.6 may be in part due to the submicron crystallite size (crystallite dimensions: a=63 error 12 nm, c=310 error 74 nm for product grown at x=4.4, a=191 error 76 nm, c=747 error 266 nm for product grown at x=3.6, a~1000 nm, c~1000 nm for products grown at x=3.4 and x=3.3) (Yilmaz et al., Microporous and Mesoporous Materials, 2004, 71: 167-175).

A second diffractometer makes use of a General Area Detector Diffraction System (GADDS). The GADDS instrument allows investigation of small specimen quantities with very high counting and particle statistics. Applications for this instrument include rapid phase analysis, small angle scattering, degree of crystallinity determinations, and characterization of preferred orientation and texture. This system can also be used in the study of macroscopic or single-crystal specimens, in determining crystal quality (Figure 3), and for performing microdiffraction measurements. Both instruments are fully supported by complete data reduction and analysis software.

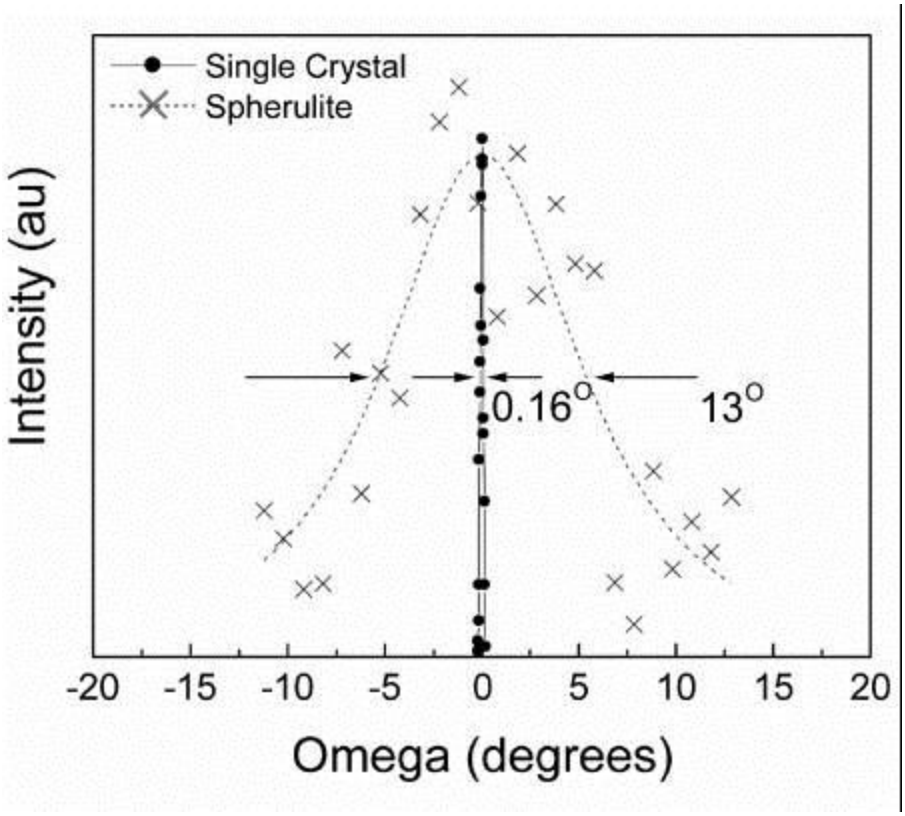

Figure 3: X-ray rocking curves for large single crystal and spherulites of ETS-4. The rocking curves were generated from the pole figures for {001} reflections and show full-width-at-half-maximum of 0.16 and 13 degrees for single crystal and spherulites, respectively, indicating a better crystal quality for ETS-4 single crystal than for ETS-4 spherulites (Miraglia et al., Journal of Crystal Growth, 2004, 270: 674-684).

The X-ray diffraction system has flexibility of vertical configuration, high-precision goniometer, and Diffrac Plus Software. Applications include quantitative phase analysis, precise lattice parameter determinations, crystallite size/strain measurements, line profile shape analysis, and total pattern fitting/structure refinement.