12. ADVANCES IN POWDER DIFFRACTION

12.1—2 SHORT TIME X-RAY POWDER DIFFRACTION USING SYNCHROTRON RADIATION. By K. Kosten and H. Arnold, Institut für Kristallographie der TH Aachen, Germany.

At HASYLAB, Hamburg, a De Wolff monochromator and a Guinier camera is installed in order to obtain diffraction patterns of phase transitions and chemical reactions. The shortest exposure time obtained was 10 sec. Low temperature devices and high temperature furnaces are available. Several phase transitions and chemical reactions have been studied. As examples the hydration of CaSO₄·1/2H₂O to gypsum and the decomposition and phase transitions of Na₂MoO₄·2H₂O are shown.

12.1—1 FILM READER PROGRAM FOR GUINIER POWDER PATTERNS. By T. Evans, M. K. Bentley, and C. M. Foris, Central Research and Development Dept., E. I. du Pont de Nemours & Company, Experimental Station, Wilmington, Delaware 19898, U.S.A.

A computer program has been developed for collecting digitized intensity/position data from x-ray powder diffraction films obtained with a Guinier-type focusing camera. This program utilizes the capabilities of the Optronics P-1700 Photomation and an Advance Electronic Design (AED) Model 512 color terminal and is operating on a Digital Electronics PDP 11/60 (RSX-11M) computer. Through a series of interactive commands the absorbance data for a specified area of the film (e.g., a narrow center strip) is read, stored and displayed. The AED 512 screen display reproduces, or artificially enhances, the film image. Both hardware and software magnification (zoom) aid cursor positioning of points to define a line, according to a least-squares fit, through the center of curvature of the reflections recorded on the film. A specified number of absorbance data values surrounding the defined line are then averaged at each vertical scan position. Finally, a disk file of averaged absorbance and film position (mm, film length) is created. This data file is used for 2θ calculation (internal standard reference) and peak-finding. The program is not specific to Guinier-type films and has been used to collect absorbance vs. position (2-dimensional) data for other types of diffraction films, such as those obtained with fibers and polymer materials.

12.1—3 THE AMORPHOUS CHARACTER AND PARTICLE SIZE DISTRIBUTIONS OF POWDERS PRODUCED WITH THE MICRONIZING MILL FOR QUANTITATIVE X-RAY POWDER DIFFRACTION. By B. R. O'Connor and Y. Chang, Department of Applied Physics, Western Australian Institute of Technology, Bentley, Western Australia.

The rapid rise in the popularity of rod micronizing mills for powder preparation in quantitative analytical x-ray powder diffraction (XRPD) has lead to the present critical examination of material produced by the method. Micronizing mill specimens of α-quartz were produced for a range of milling times under wet- and dry-grinding conditions.

Tests were conducted to infer the amorphous content of the specimens with a procedure similar to that described by Altmann-Williams, Byrnes and Jordan (Analyst (1981), 106, 69). Measured and calculated Bragg-Brentano XRPD intensities for milled α-quartz and reference corundum specimens, and measured XRPD data for material acid-washed after milling, were used to provide an estimate of amorphous content.

Particle size distributions were estimated using scanning electron microscopy (SEM) and sedimentation techniques. The results show that the micronizing mill can produce XRPD specimens of α-quartz for assay work which adequately satisfy particle size criteria, and for which negligible amorphous content is introduced by grinding.