PREFACE

Some Observations on Recent Developments in Polycrystalline Diffraction

This is a very exciting time in the field of powder diffraction. This note presents some impressions of what has occured in the last decade to transform the powder method from a mature analytical technique to a rapidly expanding and much more powerful method with many new uses and users. Several important developments came to fruition in roughly the same time frame, including the ever-expanding power and availability of computers, the profile fitting method which has greatly improved data reduction and interpretation, the Rietveld method which has become an important method for crystal structure analysis, major advances in X-ray detectors, and the use of synchrotron radiation. There were also many innovations in commercial instrumentation, particularly the rotating anode generators, but there is not sufficient space to discuss them. Previous authors in this series of Prefaces have cited a number of these developments.

An example of the important uses of the powder method is the recent discovery of the high-temperature superconductors. This involved thousands of scientists and was accompanied by world-wide publicity. The importance of the method was demonstrated to scientists who had little or no previous interest in diffraction. It immediately became the standard method to follow the progress of the solid state reactions by identifying the phases produced in the synthesis of the materials. The scientific race suddenly required powder laboratories to greatly expand their output and shorten the turn-around time. It was not unusual to operate the equipment continually and to sleep in the laboratory. Huge numbers of patterns were produced with physicists peering over the shoulders of the diffractionists to find if they had made a successful run. More advanced studies using X-rays and neutrons were made to determine the crystal structure and the site occupancy. This is a shining moment in the history of the powder method.

Profile fitting is used to determine the shape, scattering angle and width of each reflection, or a cluster of overlapping reflections, with much higher precision than was possible with the analogue strip-chart recorder method. A computer is used to fit the step-scanned data with a Lorentz, pseudo-Voigt or other appropriate curve-fitting function. The instrument function inherent in the diffractometer geometry can be precisely determined and the broadening and peak shifts arising from the specimen can be analysed. Computer graphics provide a powerful direct means of exactly comparing the observed and calculated profiles. Subtle differences in shapes become apparent and are invaluable in determining particle size, microstrain and disorder stacking.

The Rietveld method has been used for a large number of structural studies of powder samples by neutron diffraction, and it is now being used for synchrotron X-ray powder data. Combining the results of neutron and X-ray diffraction and supplemented with electron microscopy when applicable will lead to better understanding of the complex materials now being developed.

Synchrotron radiation provides a powerful new X-ray source whose properties have made possible the development of improved and new powder methods. The high intensity, parallel beam and wavelength selectively are ideal for diffraction. Time-resolved analyses with position sensitive detectors are being developed. The systematic errors inherent in the conventional focusing methods are absent in the parallel beam geometry thereby, increasing the precision of lattice parameter determination. Each reflection is a single peak 0.02° to 0.05° wide and difficulties arising from the $K\alpha$ doublet are absent. The wavelength can be selected to obtain the highest possible peak-to-background. Anomalous scattering can now be conveniently studied.

The parallel beam optics does not require the θ-2θ specimen-detector relation. This makes possible thin film analysis by depth profiling to add another dimension to the characterization. The beam enters the film at grazing incidence-less than the critical angle-and only the detector is scanned. Only the top 50 to 200Å of the film is penetrated, and the diffraction patterns from the thin surface can be compared to those of the full film thickness obtained with θ-2θ scans. The preferred orientation, inclination of selected lattice planes to the film surface and other characteristics can be determined in addition to the phases.

Synchrotron radiation is well-suited to energy dispersive diffraction because of the high intensity continuous radiation. The resolution can be increased by a factor of 10^2 to 10^3 over conventional EDD by step scanning the incident beam monochromator so that the X-ray optics rather than the detector determines the resolution; scintillation counters can be used in place of solid state detectors.

The expanded activity is shown by the increasing numbers of papers involving the use of the powder method presented at scientific meetings. The new journal Powder Diffraction began publication in 1986 and supplements the Journal of Applied Crystallography and Advances in X-Ray Analysis. The momentum generated is likely to continue and we may confidently look forward to greater use of powder diffraction.

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