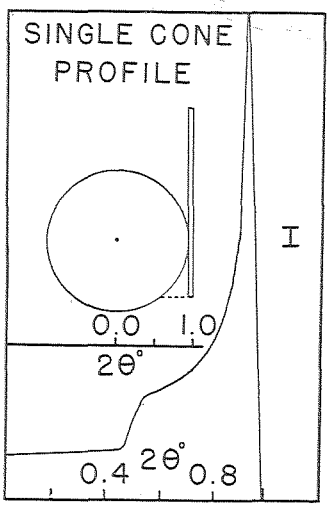
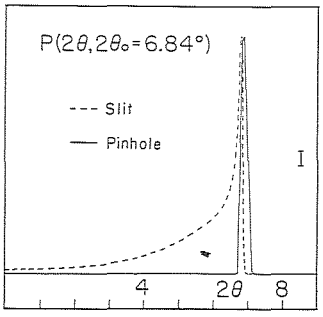
12.1-01 NUMERICAL EVALUATION OF INTENSITY ABERRATIONS. By J. H. Konnert and P. D'Antonio, Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, D. C. 20375, U.S.A.

It is important in the collection and interpretation of diffraction data to evaluate the aberrations affecting the position, shape and intensity of details in the diffraction pattern. Among contributing factors are: source size and shape; incident beam collimation; size, shape, position and absorptivity of sample; diffracted beam collimation; monochromater (if any) efficiency, and angle setting errors. For many experimental arrangements, it is not possible to express analytically the cumulative effects of these factors. However, modern computers permit the numerical evaluation of aberrations for arbitrary source, collimation and detector configurations. We have found useful a computer program that calculates for a set of experimental conditions the "profiles" that indicate the shape, position and intensity of curves that, for perfect resolution, would be delta functions. The first figure illustrates the method by which these profiles are calculated. The profile illustrated derives from a single diffraction cone of $2\theta = 1^{\circ}$ determined by a single point on the source, a single point in the sample, and a detector slit. As an insert in this figure, the geometry in the detector slit plane is also illustrated. The approximate circle represents the intersection of the diffraction cone with this plane. The detector slit may be thought of as sweeping from the center of the cone to the right as the detector ranges from $2\theta = 0^{\circ}$ to 1°. In this example, a monochromater is not present, and the observed intensity is proportional to the fraction of the cone passing through the detector slit. At 0.56°, the intensity rises abruptly as the 2nd portion of the cone also passes through the slit. At ~1°, a long cone length passes through the slit resulting in the sharp peak. Complete profiles may be computed by considering the many diffraction cones arising from finite source and sample size. While the simple geometry represented here may be expressed analytically, incident beams and diffraction cones may be evaluated numerically for the passage through any collimation system. The second figure illustrates representative profiles for slit (Kratky Camera) and approximate pinhole geometries. These low angle profiles have been chosen for illustrative purposes because of the large associated aberrations. Higher angle data will generally be affected by smaller aberrations. Recognition of such small aberrations, however, may be quite important when carrying out analyses such as profile refinements. Aberrations associated with several experimental geometries will be illustrated along with details of the numerical evaluations of the profiles.





12.1-02 AN AUTOMATED POWDER DIFFRACTION DATA REDUCTION PROGRAM FOR LARGE THROUGHPUT. By G. R. Fischer and W. T. Kane, Corning Glass Works, R&D Division, Corning, New York - 14830

A fully automated X-ray powder diffraction system at Corning Glass Works was specifically designed for large throughput of 35 or more samples per day. The required operator time is drastically reduced because functions such as sample-log keeping, running of the diffractometer and sample changing, and the numbering and labeling of the patterns and individual peaks are performed by computer. The data reduction part of the system copes automatically and without operator interaction with a large variety of materials ranging from fully amorphous to highly crystalline specimens.

Several novel approaches in the data reduction process were required to achieve the versatility needed. These include the filtering of the (fast) Fourier transform of the data set with an auto-adjusting digital filter, the approximation of the general background through the fitting of spline functions, and the automatic calculation of a variable threshold for peak acceptance based on a pseudo-standard deviation.

The program is written in a high level language (FORTRAN), easily transportable, and executes rapidly even on moderate sized minicomputers. The output consists of a table of 2θ (corr) d (corr), and I/I_1 and of a plot of the refined X-ray pattern with all peaks labeled with their corresponding d (corr) and observed relative intensities.

12.1-03 EXPERIMENTAL & COMPUTATIONAL TECHNIQUES FOR RECORDING & ANALYSING PHOTOGRAPHIC POWDER DATA. By M.J.Mendelssohn & H.J.Milledge (Crystallography Unit, Department of Geology) & D.Walley (Computer Science Department), University College London, Gower Street, London WC1E 6BT, England.

During investigations designed to optimise the parameters involved when fluorescent screens are used to register diffraction data, experiments are being made using radiation selected from the continuum (a situation which arises in both neutron and in synchrotron diffraction experiments). The tests involving powder samples are being carried out using a Stoe powder camera which permits both transmission and reflexion patterns to be recorded with monochromatised radiation.

Since the efficiency of fluorescent screens increases very rapidly as the wavelength decreases, patterns produced with radiation of ~ 0.5 - 0.7A (near the peak of the continuum) can appear to be considerably stronger than those obtainable with the Kα radiation from, for example, a Cu tube. Thus, if adequate resolution can be achieved, the continuum radiation may actually be preferable for some purposes, especially those involving real-time phenomena such as phase transitions, encapsulated specimens where hard radiation can reduce effects due to absorption, or situations where anomalous dispersion effects may be maximised by the choice of a particular wavelength, as in the Laue case (Grenville-Wells & Lonsdale, Nature (1954) 173 1145).

In order to evaluate these non-standard data sets they are being compared with conventional powder patterns obtained from the same specimens and measured with the same computer-controlled scanning microdensitometer.

The powder data are processed with a suite of PDP-11 FORTRAN programs operating in the following stages:

(1) The raw data from a number of parallel scans are presented as an intensity [I] & a standard deviation [DI] for each microdensitometer reading (usually at intervals of 0.05mm, i.e. using a 50 micron aperture).

(2) This output is inspected, and values of line intensities [I] = I(peak) - I(background), peak positions [M(obs)] in mm., and σ[M(obs)] are selected manually for both specimen and internal standard, and checked against the film itself. At present this procedure seems to be preferable to automatic peak-searching, especially in cases of very strong, very weak, or poorly-resolved patterns.

(3) Values of [M] = M(obs) corrected for the non-linearity of d^* vs. sin θ by comparison with the internal standard, [I] and σ[M] are given to an exhaustive-search indexing program which supplies possible indices for some or all of the lines.

(4) These indices are supplied to a cell-parameter refinement program, where they are cycled with an appropriate weighting scheme to remove redundant indices and to derive cell parameters and their standard deviations.

(5) Line positions [M(calc)] are generated for comparison with [M(obs)] to see whether a satisfactory match has been achieved.

(6) If accepted, the indexed data are output in any desired format and incorporated in the local databank file for use with the original data, from which information such as peak shapes and integrated intensities can be extracted later.

12.1-04 PODA: AN APPROACH TO MULTI-PURPOSE POWDER DIFFRACTION ANALYSIS, R. A. Coyle, Aeronautical Research Laboratories, Department of Defence, Melbourne, Australia.

For some time punched paper tape has been used for automatic recording of powder diffraction data. In-house computing facilities have then been used for computation and storage of the data. Paper-tape equipment is being replaced by microprocessor interfaces enabling on-line processing of some of the measurements such as searches for peaks, and residual stress. However, in an applied research laboratory, the complexity and changing nature of the tasks can be better accommodated using the capabilites of a large computer. In this context the program PODA was developed for multi-purpose analysis of powder diffraction data on a large computer. This program allows correction for both experimental and geometrical factors, calculation of the required variables, collation with previous tests and final presention of the results in graphical or tabular form.

PODA is structured like many of the DIGITAL utilities and responds to strings of characters as instructions allowing operational flexibility. The program gives simple diagnostic messages for data or instruction errors, has default options for constants and includes a facility for repetitive operators.

Some of the facilities available are to 1) read data in an any format with or without angle readings, 2) archive and retrieve data in binary form on disk or magnetic tape, 3) correct for absorption, background or Lorentz-polarization by reference to tables or by calculation, 4) eliminate alpha 2 reflections, 5) correct for dispersion, 6) calculate Fourier transforms, 7) plot pole figures, 8) fit a curve of best fit, 9) calculate centroid and area of a diffraction line, 10) hand edit data, 11) plot in a choice of formats. The standard data format includes a heading which records the operations performed on the data and the date of last change.

A number of outputs will be shown as examples, together with a copy of the operations manual.

12.1-05 A GUINIER DIFFRACTION CAMERA EMPLOYING A ROTATING ANODE X-RAY SOURCE. By J. D. Oliver and W. B. Broering, Miami Valley Laboratories, The Procter & Gamble Company, Cincinnati, OH 45247.

Rapid and reliable analytical techniques are required for prompt and effective product development. Implementation of these techniques has benefited directly from the decreasing cost and increasing availability of microcomputers.

This presentation describes a method to rapidly characterize solid-state materials at selected temperatures over the range of -100°C to +300°C. Pivotal elements of the method comprise the first successful combination of a Huber Guinier diffraction system with microprocessor-controlled variable temperature fixtures and a Rigaku RU-100 rotating anode X-ray source. Developed films are scanned with a LSI-11 automated Grant microdensitometer. High quality X-ray patterns require exposure times ranging from 5 to 12 minutes. The instrumental design and the precision of data will be described in detail.