which is related to the continuous transform of the component molecules in the crystal unit cell. Such details in e.d. patterns from anthracene taken by Charlesby, Finch and Wilman in 1939, in fact, led to the use of such information by some crystallographers for structure analysis. Generally there are two origins to this diffuse signal: thermal motion and static displacement. In polymethylene chain compounds such as the n-paraffins and polyethylene, for example, both components can be found in respective regions of the reciprocal lattice. The thermal signal is observed in the projection down the chain axes and is found to be extinguished when the samples are cooled to 40K. A much stronger diffuse pattern in the projection onto the molecular axes (epitaxially crystallized samples), however, is not diminished even when the specimen is cooled to 4 K. Models based on disordered molecular packings indicate that its origin is due to small, residual longitudinal chain translations which persist in the lamellar layers when the material is crystallized through the conformationally-disordered structure ('rotator' phase for certain chain lengths) to the lowest energy methylene subcell structure. This type of disorder fits well with the larger longitudinal chain translations observed in the reverse process, when paraffin crystals are heated toward the melting point. Diffuse scattering can also be useful for following crystal-crystal transitions in two-dimensional protein crystals. For example, the onset of a protein trimer recrystallization in hexagonal layers of the Omp F porin from *E. coli* reconstituted in DMPC was first identified by the diffuse scattering signal found in the computed Fourier transform of an electron microscope image (or seen in electron diffraction patterns). Subsequent changes in the diffuse signal, and finally its sharpening into directional streaks which then, in time, split into discrete spots, correspond to the crystallization of a polar orthorhombic cell in the less anisotropic hexagonal layer. Cross-correlation analysis on images also indicates that the total sampled area can contain recrystallized orthorhombic subareas oriented in any of three possible directions. This is the first direct observation of cooperative rotational diffusion of a membrane protein in a lipid bilayer.

PS–02.05.07 THE DISORDERED STRUCTURE OF MULLITE USING DIFFUSE X-RAY SCATTERING. By B. D. Butler*, T. R. Welberry, and R. L. Withers, Research School of Chemistry, Australian National University, Canberra A.C.T. 0200, Australia

A description of the three dimensional disordered arrangement of oxygen vacancies in a mullite of composition $Al_2(Al_{2+2x}Si_{2-2x})O_{10-x}$ where x=0.4 has been developed which is consistent with the measured diffraction data. The structure of this material was modelled using Monte Carlo techniques where the oxygen vacancies were allowed to interact via a set of pair energies. Cations in adjacent tetrahedrally coordinated sites were given displacements that depended only on the local arrangement of these oxygen vacancies. A calculation of the diffraction pattern from this model crystal compared favourably with measured diffraction data. Not only can this model describe the origin of the observed incommensurate diffraction maxima in the $2c^*$ reciprocal section but it is also consistent with many broader diffuse diffraction features that have been observed in other reciprocal sections. In addition to large repulsive $2\cdot110\grave{O}$ and [110] interactions that are required to satisfy certain cation bonding requirements, it was found that unequal vacancy repulsive interactions were required along $\cdot100\grave{O}$ and $\cdot010\grave{O}$. Attractive vacancy–vacancy interactions along $2\cdot112\grave{O}$ and $\cdot001\grave{O}$ were also necessary but in the latter case the magnitude of the interaction is such that the probability of having an $\cdot001\grave{O}$ vacancy pair was near that of a random vacancy distribution.

PS–02.05.08 ATOMIC FORCE MICROSCOPY HELPS DOMAIN STRUCTURE DETERMINATIONS BY X-RAY AND THERMAL ANALYSES. By N. Masaki*, Fac. of Pharm. Sci., Kyoto Univ., Kyoto, Japan, and Y. Yoshimura, Fac. of Sci. & Eng., Ritsumeikan Univ., Kyoto, Japan, and H. Kado, K. Yokoyama and T. Tohda, Central Res. Labs., Matsushita Elec. & Ind. Co. Ltd., Moriguchi, Japan, and N. Man and M. Sakiyama, Fac. Sci., Osaka Univ., Toyonaka, Japan.

Crystal structure of phenytoin (diphenyl hydantoin $C_3N_2H_2O_2 \cdot 2(C_6H_5)$, a=6.230, b=13.581, c=15.532 Å, $Pn2_1a$, Z=4) of famous anticonvulsant drug for epilepsie had been determined at room temperature by Camerman & Camerman[1]. We found a reversible phase transition of the crystal of phenytoin at 183.5°K by X-ray diffraction photographs and confirmed by thermal analysis. Satellite reflections in diffraction pattern and λ-shaped curve of thermal analysis convinces of that it is a typical order-disorder phase transition caused by domain structure formation in lower temperature phase. Atomic force microscopy (AFM) revealed the domain structure even at room temperature. Therefore domain structure is inherent in phenytoin crystals through across the phase transition temperauture. X-ray pattern in high temperature phase can not detect the phase boundary. This is the third application of AFM for X-ray analysis followed structure determination[2] and space group determination[3]. New technique of domain structure analysis by X-ray diffraction and AFM will be discussed.

[1] Camerman, A. & Camerman, N. (1971). Acta Cryst. B27, 2205.
[2] Masaki, N. et al. (1992) Ultramicroscopy, 42-44, 1148. Chem. Pharm. Bull. 39, 1899.
[3] Masaki, N. et al. (1992) Asian Crystallographic Association Conference Abstract, 14s31 (Singapore, Nov.)

PS–02.05.09 MEASUREMENT AND ANALYSIS OF X-RAY DIFFUSE SCATTERING FROM PROTEIN CRYSTALS. By Bin Yu, Donald L. D. Caspar, Youli Li, Rosenstiel Basic Medical Research Center, Brandeis University, Waltham, MA 02254–9110, U.S.A.

Analysis of diffuse X-ray scattering provides important information about correlations of atomic movements in protein crystals. Computer modeling of such correlations, either based on analytical considerations or empirical observation, can be tested by comparison with 3–D diffuse scattering pattern. Data taken from tetrahedral lysozyme crystals on synchrotron at Brookhaven National Laboratory show strong modulations in its diffuse scattering patterns. We are conducting experiments to find differences in diffuse scattering resulting

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from different localized disorder in cubic insulin crystals equilibrated at different pH and salt concentrations. We have collected data on film using laborotary source, collecting 3–D data with imaging plate on synchrotron is being planned. Collecting data with good statistics is the main challenge in such studies since intensity of diffuse scattering is about 0.1 to 1 percent that of Bragg reflections and many factors contribute to scattering in background. Extraneous scattering from air, guarding slits etc. must be minimized to ensure a low and uniform background. A high order to order resolution (i.e. at least five times that of the lattice spacing), a well collimated monochromatic X–ray beam and a well characterized detector are necessary to accurately record the haloes surrounding Bragg peaks.

PS–02.05.10 EMPIRICAL AND CALCULATED THERMAL-DIFFUSE-SCATTERING CORRECTIONS FOR SINGLE-CRYSTAL DIFFRACTION DATA COLLECTED WITH A TWO-DIMENSIONAL POSITION-SENSITIVE DETECTOR. By G.J. McIntyre*, Institut Laue-Langevin, B.P. 156, 38042 Grenoble Cedex 9, France.

If the resolution of the diffractometer is assumed to be infinitely small the amount of one-phonon thermal-diffuse scattering (TDS) included in the scan through a Bragg reflection is directly proportional to the radius of the peak integration volume, the amount of two-phonon TDS to the square of the radius, and the amount of incoherent (flat) background to the cube of the radius. These differences in the dependence on the size of the integration volume can be exploited to correct for TDS and to estimate the elastic constants empirically, provided each reflection is sampled in three dimensions, as in scans made with a two-dimensional position-sensitive detector.
The TDS corrections for intensities derived by summation of counts in three dimensions are discussed in detail. The precision in the empirical method is poor for weak reflections, but, because of the slow variation of TDS with the scattering vector, the corrections for these reflections can be estimated from those of nearby strong reflections.
One advantage offered by position-sensitive detectors is optimal delineation of peak and background to minimise the estimated error in the background-corrected integrated intensity. For weak reflections this might imply integration within an envelope smaller than the instrumental resolution volume. The possible errors in the correction in this circumstance and for neighbouring reciprocal lattice points are estimated.

02.06 – Computer Graphics in Crystallography

PS–02.06.01 THE APPLICATION OF GRAPHIC DESKTOP SOFTWARE IN SINGLE CRYSTAL DIFFRACTOMETRY. By D. Abeln* and J. Kopf; Institut für Anorganische und Angewandte Chemie der Universität Hamburg, Martin-Luther-King-Pl. 6, D-20146 Hamburg, FRG.

One of the first scientific instruments to be controlled by a computer was the single crystal diffractometer (Busing, W. R. and Levy, H. A., Acta Cryst., 1967, **22**, 457). Early computer-controlled diffractometers were built at Hilger & Watts (Y290), Enraf-Nonius (CAD4), Philips (PW1000) and Siemens (AED). The programs developed for those instruments were written in assembler, mostly for a DEC PDP-8 computer. The first diffractometer software, completely written in the high-level computer-language FORTRAN IV, was the control program for the Syntex $P2_1$.

The past ten years have seen a revolution in computing and graphics hardware with the arrival of PCs and powerful graphic workstations which become increasingly faster and cheaper. New desktop systems, like GEM, WINDOWS or X/WINDOWS, allow an unexperienced user easy interaction with the computer.

In connection with the electronic rebuilding of a 22 years old, mechanically still reliable Hilger & Watts (Y290) we have developed a new graphically oriented program for the diffractometer control which uses the above mentioned advantages. A new interface (Lange, J. and Burzlaff, H., J. Appl. Cryst., 1991, **24**, 190), using a 68008-based single-board microcomputer for serving the four stepper motors of the four circles, is connected to an Atari Mega ST2 *via* the serial interface RS232. The diffractometer control software is completely written in FORTRAN77 and has the following features:

- drop-down-menus, dialog-, alert- and fileselector–boxes
- fast random peakfinding routine
- rotation- and axes-photographs
- centering routine with graphical representation of reflection profiles
- flexible indexing-, lsq- and bravais-routines
- graphical simulation of precession photographs
- ψ-scan of single reflections with transmission curve
- flexible data- and ψ-data-collection output in windows

The power of this new program Y290 is derived from a sophisticated menu-driven user interface which is much easier to use than the "classical" command-line input.

PS–02.06.02 VISUALISATION OF CRYSTALLOGRAPHIC DATA USING INTERACTIVE COLOUR GRAPHICS. K.M.Crennell*, ISIS Instrumentation Division, Rutherford Appleton Laboratory, UK

Facilities are described for the visualisation of data at the pulsed neutron source, ISIS, at the Rutherford Appleton Laboratory in the UK, where there are many instruments used to determine the molecular and crystalline structure of materials at a wide range of temperature and pressure. Programs have been written to combine data collected at a series of different operating conditions into a single multi dimensional data set, which can be visualised using image processing manipulation techniques. Examples are given of improved observation of magnetic phase changes at temperatures near absolute zero.

ISIS data collection uses a VAX cluster; most of the colour graphics is made by the UNIRAS package which can display data in 1, 2 or 3 dimensions interactively using X-terminals, or send hardcopy to PostScript printers. Examples are shown of the use of colour graphics to display data as an isometric suface and during data taking, to both monitor instrument performance and improve the quality of data collected.

Following data refinement, molecular and crystal structures need to be displayed. Examples of output made using personal computers are shown. These are becoming both cheaper and more powerful and can be a more effective display tool than a remotely connected terminal displaying graphics over a busy network.

PS–02.06.03 MOLDRAW: ADVANCED MOLECULAR GRAPHICS ON A PERSONAL COMPUTER

P. Ugliengo+, G. Chiari* and D. Viterbo+

+Dipartimemto di Chimica Inorganica, Chimica Fisica e Chimica dei Materiali, Università, Via P. Giuria 7, I-10125 Torino, Italy.
*Diparimento di Scienze Mineralogiche e Petrologiche, Università, Via Valperga Caluso 37, I-10125 Torino, Italy