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

