02-Methods for Structure Determination and Analysis, Computing and Graphics

We developed the CRISP image processing system for electron crystallography, in order to make it possible for any EM laboratory to set up electron crystallography. HREM images are digitized by a standard CCD camera and processed by CRISP. The FT of the image (256 x 256 pixels) is calculated on a personal computer in only 4 seconds. From the FT the defocus, crystal tilt and resolution may be estimated. The lattice is indexed and refined and amplitudes and phases are extracted from the FT for all diffracted beams. The crystal symmetry can be determined, even for quite thick and/or misaligned crystals. The correct symmetry can be imposed, remarkably improving the projected potential map. Finally metal atom coordinates in oxides are determined with a precision of typically 0.1 Ångström.

Recently we have also developed a system, called ELD, for extracting electron diffraction intensities from film (Zou, Sukharev and Hovmöller, Ultramicroscopy 49 (1993) in press).

MS-02.04.05

SOLVING CRYSTAL STRUCTURES FROM ELECTRON CRYSTALLOGRAPHY DATA *VIA* MAXIMUM ENTROPY AND LIKELIHOOD

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The maximum entropy-likelihood method as formulated by Bricogne (Bricogne, G. *Acta Cryst.* (1984) **A40,** 410-445), and implemented by Bricogne and Gilmore (*Acta Cryst.* (1990) **A46,** 284-297) provides a powerful phasing technique for use in the electron crystallography of small molecules. We report here the process of entropy maximisation and likelihood evaluation coupled with the building of phasing trees to solve such problems.

The maximum entropy (ME) method is ideal in these circumstances because:
(1) It will work with projection data.
(2) It is stable regardless of data resolution.
(3) It uses non-uniform atomic distributions which are continually updated in the light of new phase information.
(4) It is not sensitive to data errors: this is important here where dynmical effects can produce systematic errors in intensity mesurements.

We have applied the technique to both organics and inorganics including:

(a) $CuCl_2.3Cu(OH)_2$ (Voronova, A.A and Vainshtein, B.K. (1958) *Sov. Phys. Crystallogr* **3,** 445-451)
(b) Polyethylene. (Dorset, D.L. *Macromolecules* (1991) **24,** 1175-1178)
(c) Diketopiperazine (Dorset, D.L., *Acta Cryst.* (1991) **A47,** 510-515
(d) Poly(ε-caprolactone) (Dorset D.L., *Proc. Natl. Acad. Sci. USA* (1991) **88,** 5499-5502)
(e) Copper perchlorophthalocyanine (Dorset, D.L., *Ultramicroscopy*, (1991) **38,** 41-45)
(f) Polybut-1-ene.
(g) γ-poly(pivalolactone) (Dorset, D.L., *Macromolecules*, (1992) **25,** 4425-4430)
(g) Perylene (Donaldson, D.M., Robertson, J.M. & White, J.G. *Proc. Roy. Soc. (A)* (1953) 220, 311-321) in projection using 21 reflections in total.

In all cases the method has proved to work routinely.

MS-02.04.06 **STRUCTURE FACTOR AMPLITUDE AND PHASE DETERMINATION BY OMEGA ENERGY FILTERED CBED.** J. Mayer, C. Deininger and G. Necker, Max-Planck-Institut für Metallforschung, Stuttgart, Germany.

Convergent beam electron diffraction (CBED) makes possible to obtain dynamical diffraction data from very small crystal areas (typically a few nanometers). Accurate quantification of the intensities, however, requires that energy filtered data are used because only the elastically scattered electrons contain the desired crystallographic information. Recently, imaging energy filters have become commercially available and energy filtered diffraction patterns can now be recorded without scanning using efficient parallel (2-dimensional) detection. We have used a Zeiss EM 912 Omega equipped with a 1024x1024 slow scan CCD camera to record energy filtered CBED patterns. The quantification of the intensity data is based on many beam dynamical calculations which have to be repeated over a wide range of parameter values in order to refine the structure factor amplitudes and phases. We have modified the original method by Zuo and Spence (Ultramicroscopy 35, 1991, 185-196) to make use of the two-dimensional nature of the energy filtered patterns and to improve the efficiency of the refinement process. Based on a fast computer complete sets of low order structure factors can be obtained from the crystals under investigation. Structure factor phases can be measured with an accuracy of about one degree. BeO will be discussed as an example (J.M. Zuo, J.C.H. Spence, J. Downs and J. Mayer, Acta Cryst., to be published). From a whole set of low order structure factors maps of the charge density distribution in the crystal can be determined and related to the properties of the material. The method is so far restricted to inorganic crystals of known structure but extensions to large unit cells or unknown structures are possible and will be discussed.

MS-02.04.07 USE OF COHERENT ELECTRON DIFFRACTION AND CONICAL SCANNING IN OBTAINING DATA FOR DIRECT PHASING OF REFLECTIONS IN ELECTRON DIFFRACTION. by J.W.Steeds and R.Vincent, Physics Department, University of Bistol, Bristol BS8 1TL,UK

The influence of dynamical scattering on electron diffraction data may be reduced by concentrating on higher order Laue zone (HOLZ) diffraction in convergent beam electron diffraction (CBED) data. Data obtained in this way has been used both to refine atomic parameters with high accuracy and to solve structures by use of conditional Patterson transforms. As yet, only simple version of direct methods for phasing have been applied to limited data sets of intense reflections related by closed vector loops in centro-symmetric crystals (phase triplets with net zero phase). In an attempt to reduce non-systematic

