

14.4-2 A STUDY OF DEPARTURE FROM PROJECTION APPROXIMATION IN CuAsS/Se FOR CBED & HREM. By P. Goodman, Division of Chemical Physics, CSIRO, Australia, and A. Olsen, Physics Department, University of Oslo, Norway.

The "tilt effect", or departure from a "projection approximation" (P.A.) with rotation from a zone setting has been examined during a study of CuAsS/Se (Whitfield, H.J. (1981) J. Sol. Stat. Chem. 39, 209). This effect is an important component of CBED & HREM techniques:

In CBED space-group analysis the tilt effect is utilised, when Laue-circle rather than zone-axis data is used to find the central-plane (C-P, or reciprocity) symmetries. Here the C.P.A. (C-P-approximation) is invoked.

Use of HREM for symmetry analysis has been restricted by the sensitivity of images to fractional-degree tilts from exact alignment. In combined CBED-HREM studies it is useful to know how the experimental conditions of the two techniques come into the tilt effect.

An ensemble of computed lattice images made around the [100] zone of CuAsS/Se (Pbcn) for a 9 mrad. radius has allowed the influence of the 3-dimensional structure to be examined, and for experimental images to be usefully indexed.

Conclusions relating to the symmetries generated by the vertical glide plane are as follows: With single axis rotations around the plane normal, the image retains a mirror symmetry. By contrast the CBED pattern retains this symmetry only within the 000 disk, (C.P.A. rules), in marked departure from the simple Ishizuka (Ishizuka, K. (1982), 40th. Proc. E. M.S.A., 688) rules for translational symmetries.

14.4-3 HIGH RESOLUTION ELECTRON MICROSCOPY AND IMAGE SIMULATIONS OF COINCIDENCE SITE PATTERNS DUE TO OVERLAP OF VARIANTS IN Au<sub>2</sub>Mn. By W. Coene<sup>1,2</sup>, D. Van Dyck<sup>2</sup>, G. Van Tendeloo<sup>2</sup> and J. Van Landuyt<sup>2</sup>. /1/ Research Assistant for the Belgian Fund for Scientific Research. /2/ University of Antwerp, RUCA, Groenenborgerlaan 171, B-2020 Antwerp, Belgium.

The reduction of symmetry associated with the ordering of Au<sub>2</sub>Mn gives rise to six orientation variants. High resolution electron micrographs sometimes contain images of crystal parts whereby two coaxial variants with the common axis along the electron beam, are separated by a contact plane which is inclined with respect to the electron beam. These micrographs reveal typical image reinforcements giving rise to dot patterns which are related to the coincidence lattice of the structure in both domains. A first intuitive interpretation could be based on the assumption that the bright dots correspond to columns of the Mn atoms, continuous and non-interrupted across the interface. Unambiguous conclusions can only be drawn by computer simulations using reliable structure models. However, because of the extended overlap region between both domains, the complete dynamical calculations require such a large number of diffraction beams that conventional algorithms such as the multislice method, are inappropriate. The real space patching method on the other hand is not limited by the computer memory and has a favourable calculation speed especially when extended areas are concerned. This method is not only suitable for dynamical calculations at isolated defects, since it can avoid the use of the periodic continuation technique, but also for the calculation of large unit cells and complex configurations. The first results clearly show the suggested one-to-one correspondence between the complete Mn columns and the white dots; moreover, a parameter sensitive fine structure appears in between the dots, which contains relevant information about the overlap configuration.

14.4-4 THE IMAGING OF MOLECULARLY CLEAN SURFACES IN BFTEM. By C.E. Warble, CSIRO, Division of Chemical Physics, P.O. Box 160, Clayton, Victoria, Australia, 3168.

Bright-field transmission electron microscopy (BFTEM) provides a useful means for the direct observation of materials surfaces at sub-nanometer resolutions in both "static" and "dynamic" situations. The use of BFTEM generally in the study of point defects (vacancies and interstitials), line defects (dislocations), planar faults (stacking) and twinning is well established. With few exceptions, the practice and technique of studying materials under circumstances allowing direct observation of molecularly clean surfaces in BFTEM is not so widely utilized. J.M. Cowley (Ann. Rev. Phys. Chem., 29, 1978, 251), and K. Yagi, K. Takayanagi and G. Honjo (Crystals, Growth, Properties and Applications, 7, Springer-Verlag Berlin, Heidelberg, 1982, 47) provide good recent reviews for electron microscopy surface studies.

The author's work has relied primarily upon a Hitachi HU-125 with conventional hairpin filaments and incorporating a specially designed specimen stage and holder (Mills, J.C., and Moodie, A.F., Rev. Sci. Instr. 39, 1968, 962, and Warble, E.C., Rev. Sci. Instr. 54, 1983, 607). Surface step structure in the 4-8 Å range was first directly observed in BFTEM in 1967 (Moodie, A.F., and Warble, C.E., Phil. Mag., 16, 1967, 891), and many of the classical features of step growth at the unit cell level in 1971 (Moodie, A.F., and Warble, C.E., J. Cryst. Growth 10, 1971, 26). Some applications of this type of study to solid reactions and sintering were discussed in 1969 (Stringer, R.L., Warble, C.E., and Williams, L.S., Materials Science Research, Vol. 4, Plenum Press, 53) and 1975 (Moodie, A.F., and Warble, C.E., Sintering and Catalysis, Plenum Press, 1). More recent work has utilized the JEOL 200CX with LaB<sub>6</sub> filaments and standard top entry stage.

Whether or not UHV environment and/or special cold/hot specimen stages are utilized, the following general conditions have been found to maximize the possibility of directly observing surface step structure of sub-nanometer dimensions.

- (1) Cleanliness  
Everything that sees the microscope vacuum must have minimal contamination.
- (2) Specimen support  
Avoid random phase screen effect of amorphous films.
- (3) Minimum size effective source  
Increased illumination coherence leads to high contrast in phase contrast images.
- (4) Defect of focus  
500-1000 Å underfocus in microscopes with spherical aberration constants of 1-2 mm.
- (5) Best possible vacuum  
Hitachi HU-125 gun pressure  $2 \times 10^{-7}$  Torr  
specimen area approx.  $10^{-9}$   
JEOL 200CX column pressure  $7 \times 10^{-8}$  Torr.
- (6) Sufficient resolution
- (7) Freedom from water vapour  
Particularly critical in hot stage microscopy.

While all seven are important, and best results are obtained with simultaneous optimization, the first four are most critical to the observation of surface fine structure in BFTEM.

Examples will be shown of both surface structure, and the combined imaging of lattice and surface step structure in such materials as MgO, Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub>.