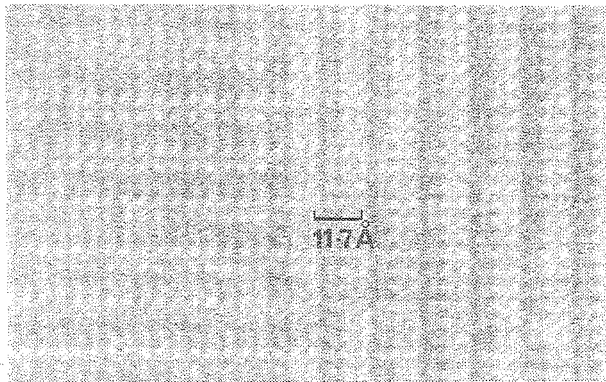
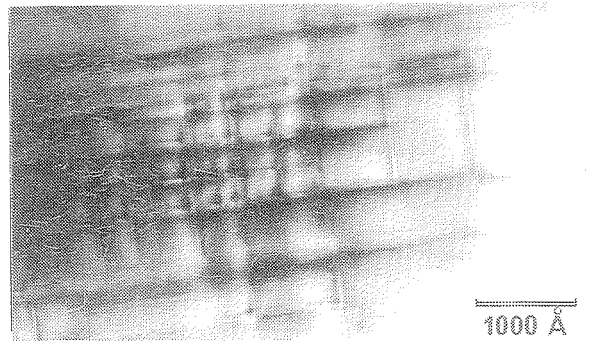


16.X-1 SINGLE CRYSTAL DIFFRACTOMETER DATA.

By T. A. Hamor, University of Birmingham, Birmingham, England, H. Steinfink, The University of Texas, Austin, TX, U.S.A., and B.T.M. Willis, University of Oxford, Oxford, England.

The wide spread use of the computer controlled single crystal diffractometer and associated software packages frequently permit the solution of unknown crystal structures within days after the initiation of the procedure. The main interest may lie in the determination of the molecular conformation and the accuracy of bond lengths or thermal vibrational parameters may not be paramount. However, having done the analysis, the wish to make the results part of the scientific literature is irresistible. Acta Crystallographica recognizes the varying driving forces but, unless minimum standards are met, erroneous results will creep into print. The quality of the final result is determined by care spent on the data collection. The length of time devoted to this step represents a compromise between the desire to achieve excellence and the demand for the instrument by many scientists in an organization. This presentation discusses the minimum requirements which will ensure the correct choice of the unit cell, accurate cell constants, the various scan modes for collecting accurate intensities and the extent to which reciprocal space needs to be explored. The latter has a critical bearing on the choice of the correct space group and the use of Friedel and Bijvoet pairs for the structure determination. The extraction of the corrected intensities from the measured data is not trivial if defects, disorder, thermal diffuse scattering, etc. are present in the crystal. These problems will be addressed.

LATTICE IMAGE OF MICROSTRUCTURE IN $Ba_3La_3Cu_6O_{14}$ LOW MAGNETIC MICROGRAPH OF MICROSTRUCTURE IN $Ba_3YCu_3O_7$

16.X-2 MICROSTRUCTURE IN HIGH TEMPERATURE SUPERCONDUCTORS OF PEROVSKITE TYPE OBSERVED IN HIGH RESOLUTION ELECTRON MICROSCOPY AND IN SITU CONVERGENT BEAM DIFFRACTION. By A.F. Moodie and H.J. Whitfield, CSIRO Division of Materials Science and Technology, Locked Bag 33, Clayton, Victoria 3168, Australia.

High temperature superconductors of both the lanthanum and yttrium series have been prepared and examined using high resolution electron microscopy and in situ convergent beam electron diffraction.

Space groups have been determined and lattices have been imaged at the 2\AA level for comparison with multislice calculations.

An extensive microstructure has been found to be associated with apparent single crystals in both series of compounds. Apart from a few isolated dislocations and Wadsley defects the microstructure in the tetragonal phase of the lanthanum series consists of substantially strain-free three-dimensional intergrowths, sometimes exhibiting variability in the number of cubic units along the c-axis. Intergrowth in the orthorhombic phases of the yttrium series, on the other hand, is associated with appreciable strain.

Details of the intergrowths will be presented, and comparisons of observed and calculated intensity distributions will be made.

16.2-1 THE DELTA-DIFFRACTOMETER - A NEW TYPE OF FOUR-CIRCLE-DIFFRACTOMETER. By H. Burzlaff, J. Lange and W. Rothammel, Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Universität Erlangen-Nürnberg, Bismarckstr. 10, 8520 Erlangen, FRG.

The principles of construction of a new four-circle diffractometer will be presented. The instrument consists of a classical concentric pair of omega/2-theta-axes in vertical arrangement with two additional axes. The 2-theta-circle carries a horizontal axis for a new circle that allows a second independent motion for the counter device; the omega-circle carries an additional delta-circle for a second independent motion of the crystal, the delta-axis being inclined at 30 degrees with respect to the omega-axis. All axes intersect at the position of the crystal.

The advantages of this arrangement are

- (i) new and faster procedures for the alignment of crystal and instrument;
- (ii) free space for the installation of additional equipment such as low temperature devices etc.
- (iii) new techniques for measurements.

Disadvantages are

- (i) some restrictions with respect to certain regions of reciprocal space;
- (ii) more complicated reflection conditions as the equatorial plane of the Ewald sphere must be left.

The device works under control of a LSI 11/23 computer. First results and experiences with the instrument will be reported. Moreover a new procedure for absorption corrections will be presented. The new instrument allows the reconstruction of the scattering crystal volume while the correction data are derived from absorption shadows in the primary beam. The correction procedure will be incorporated in the normal measuring routines.