

16.X-01 SEMICONDUCTOR-CONDUCTOR TRANSFORMATION IN GaAs AS A PRESSURE-FIXED POINT. By S. Akimoto, Institute for Solid State Physics, University of Tokyo, Minato-ku, Tokyo, Japan.

As an official activity of the Commission on Crystallographic Studies at Controlled Pressures and Temperatures, the extension of the study of transformations at specific pressures to the higher pressure region was undertaken. The target was focused to the semiconductor-conductor transformation in GaAs which occurs in the region 180-193 kbar according to previous investigations. This is a direct extension of the former report on the transformation pressure in ZnS by Block at the Warsaw meeting (1978).

A high-purity, non-doped crystal of GaAs was distributed to more than ten laboratories as a standard sample. In the participating laboratories, the transformation was detected by X-ray diffraction, electrical resistance as well as visual observation, and the transformation pressure was determined based on the compression of NaCl and the shift of the ruby R_1 fluorescence line. Both the diamond-anvil cell and the multi-anvil type high-pressure apparatus were used for the present round-robin study. The most probable value of the semiconductor-conductor transformation pressure in GaAs is reported based on the data presented by the participating laboratories. The correlation of the transformation pressure obtained by the different methods and by the different high-pressure apparatus is also shown.

16.X-02 A COMPARATIVE STUDY OF THE CHARACTERISTICS OF X-RAY FILMS. By S.C. Dawson, O.S. Mills and M. Elder, Department of Chemistry, University of Manchester, Manchester, M13 9PL, and Science Research Council, Daresbury Laboratory, Warrington, WA4 4AD, U.K.

X-ray film characteristics, last surveyed by Morimoto and Uyeda (Acta Cryst. 16, 1107, 1963), have been reinvestigated using 23 available X-ray films. The properties which have been measured include thickness and silver content, speed, granularity at four different optical densities, background fog level, film factor, and linearity and uniformity of response. The X-ray measurements were made using Ni-filtered Cu fluorescent radiation, exposing packs of two films to record rows of spots of regularly stepped intensity. All the films were processed using standard chemicals for comparison purposes, and where the manufacturers issued specific recommendations for chemicals additional films were processed using these materials. Digitised density data were obtained with an Optronics drum scanner and checked with a Joyce-Loebl machine. The effect of ageing has also been assessed. The 23 films exhibited relative speeds ranging from about 1.4 to 0.1 compared with the now unavailable Ilford Industrial G at 1.0.

16.X-03 COMPARISON BETWEEN FILM AND DIFFRACTOMETER DATA. By Oliver Lindqvist and Astrid Magnusson, Department of Inorganic Chemistry, Chalmers University of Technology and the University of Göteborg, S-41296 Göteborg, Sweden

The IUCr Microdensitometer project, Part I: Inter-Experimental Agreement (J. Appl. Cryst. (1980) 13, 318-337) compared the precision of film intensity measurements performed at fifteen different laboratories. The aim of the second part of the project is to obtain an estimate of the accuracy of microdensitometer intensity data. The study comprises two parts:

1) Collection of three-dimensional data on precession films and with a four-circle diffractometer from the smaller of the sodium tartrate dihydrate crystals used in Part I of the project. Comparison of the refined structural parameters and evaluation of the relevance of the standard deviations of these parameters with respect to different estimates of $\sigma(I)$ for film data. The previous diffractometer study (Ambady, G.K. & Kartha, G. Acta Cryst. (1968) B24, 71-83) will also be included.

2) Refinement of the ab projection of the sodium tartrate dihydrate based on each of the fifteen hk0 data sets submitted by the participants in the microdensitometer project. Analysis of the parametric spread with respect to the standard deviations in parameters obtained for different intensity weighting systems, i.e. based on the $\sigma(I)$ values supplied by the participants and on artificial weights.

16.X-04 PROTEIN CRYSTAL OSCILLATION FILM DATA PROCESSING: A COMPARATIVE STUDY. J.R. Helliwell, A. Achari, A.C. Bloomer, P.E. Bourne, P. Carr, G.A. Clegg, R. Cooper, M. Elder, T.J. Greenhough, B. Shaanan, J.M.A. Smith, D.I. Stuart, E.A. Stura, R. Todd, K.S. Wilson, A.J. Wonacott, P.A. Machin.

On behalf of the UK Collaborative Computational Project for Protein Crystallography SRC, Daresbury Laboratory, Warrington WA4 4AD, England.

A project has been instigated to evaluate the relative performance of different microdensitometers and protein crystal oscillation film data processing packages used in the U.K. The single crystal oscillation data set chosen for study was from a horse spleen apoferritin crystal, space group F432 (cubic with $a = 184.0 \text{ \AA}$, with X-ray data collected on a conventional source at Cu K α wavelength (1.5418 Å). The particular advantages of this crystal space group system were that only 15° total rotation angle was needed for a complete data set (neglecting a blind region) with four equivalent reflections which could be collected from a single crystal before serious radiation damage of the sample. Hence, by avoiding problems of crystal to crystal scaling, comparisons concentrate on the film scanning and processing methods and with a relatively small computing effort finally arrive at a merged data set with an R sym in each case. Three different types of scanners, the Joyce-Loebl Scandig-3 (used at 50 μm and 100 μm raster) the Optronics Photoscan (100 μm raster) and a flying spot densitometer are being compared. In total, four Scandigs and three Optronics instruments are being separately used at the various institutions involved in this project. The separate data processing software packages involved utilize both off-line and on-line methods.

Results are presented which compare film scanners (at 50 and 100 micron resolution), orientation matrix calculations, reflection integration and film to film scaling techniques by appropriate reference to quantities such as least squares residuals, R-factors on intensity in each case and an analysis of the agreement of the results of the different methods.

But the original flat two-dimensional type of multi-wire counter which Charpak had developed in 1968 (together with an x,y position readout scheme using delay lines devised by Perez-Mendez at UC Berkeley) is the basis of the MARK I detector we have been using in our lab in San Diego for 6 years. This detector has collected the data used to solve 4 new protein structures.

Two other area diffractometer systems using single, flat two-dimensional MWPC's are now in the beginning stages of operation. One of these is being assembled at the Univ. of Virginia. The detector has many features in common with our MARK I detector including delay line type position readout but its delay lines are faster for reduced dead time losses. Another system using a single flat multiwire counter is being assembled at the Stanford synchrotron. The detector in this system includes two very fast printed circuit delay lines needed to speed up the x,y position readout and allow counting at the high rates anticipated at the synchrotron. Both the Virginia and Stanford groups expect to collect some protein data by this summer.

In our laboratory in San Diego, a second generation area diffractometer system is now in operation. This system, called MARK II, is designed to accommodate as many as ten flat area detectors of an improved type designed especially for use in detector arrays and having better energy resolution and higher counting efficiency than our MARK I detector. Two of these detectors are now in operation and two more will be in operation late this year. The X-ray source for this new system is an Elliott rotating anode with monochromator and beam intensity monitor. The crystal-to-detector distances are adjustable from 30 cm to 2 m and the detectors can be positioned to approximate the surface of a sphere centered at the crystal. Work with 400 Å unit cells should be possible at speeds at least 200 times faster than a standard diffractometer.

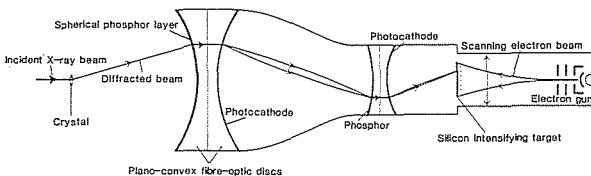
16.X-05 STATUS OF MWPC AREA DIFFRACTOMETERS. R. C. Hamlin and Ng.-h. Kuong, Depts of Physics, Chemistry and Biology, Univ. of Calif., San Diego, La Jolla, CA 92093 USA.

Diffraction from crystals of large biological molecules consists of many simultaneous reflections with small angular separation. To measure this radiation efficiently a photon-counting detector is required which intercepts a large solid angle and can independently count each of the diffracted beams which strike it. Ideally this detector would intercept the whole solid angle containing diffracted beams so that no diffracted radiation would be lost and the most possible information would be obtained from a single crystal.

Several types of detectors were proposed to meet this need. Arndt in 1966 proposed that a high-gain television detector could be used and he and others have pursued this idea but a television system has not yet been used to solve any protein structures. Georges Charpak in 1968 reported his development of the flat multiwire proportional counter (MWPC) and pointed out that this type of detector would be quite useful for detecting x-rays. In 1974 Charpak reported a more complex type of MWPC with a "spherical drift" region fitted on front which he had designed specifically for detecting x-rays. Two 50 cm x 50 cm detectors based on this design were built at CERN. One was sent to LURE at Orsay, France and the other to MIT in the United States. The groups at these two laboratories have tested these spherical drift chambers and shown them to be suitable for large-molecule crystallographic work. The MIT group has collected a small amount of data from Ribonuclease A ($P2_1$, $a=30$ Å, $b=30$ Å, $c=50$ Å) a strong diffractor and reports intensity R factors in the 11-12% range. They are working on further instrument calibrations to improve this measurement accuracy.

16.X-06 HARDWARE AND DATA-COLLECTION SOFTWARE OF A TV AREA-DIFFRACTOMETER. By D.J. Thomas and U.W. Arndt, MRC Laboratory of Molecular Biology, Hills Road, Cambridge, CB2 2QH, England.

A strategy to collect diffraction data efficiently and accurately with an area-detector is established, and the software developed to collect such data at high speed on an area-detector diffractometer using normal beam rotation geometry is described.



The general design criteria of television detectors (U.W. Arndt & D.J. Thomas, Proc. ESF Workshop on X-ray position sensitive detectors and energy discriminating detectors (Hamburg, 1980), Nuc. Inst. & Methods: in press), our hardware (U.W. Arndt & D.J. Thomas, *ibid.*) and a method to reduce the systematic digitisation errors in collected data (D.J. Thomas, *ibid.*) have been described.

The software (D.J. Thomas, *ibid.*) used to predict at high speed the order of occurrence of diffraction spots over any part of the surface of an area-detector involves the use of unconventional lattice indices which bear a resemblance to cylindrical polar coordinates, some new algorithms in lattice theory (D.J. Thomas, Acta Cryst.: in press) and a "real-time" adaptive refinement routine to monitor the condition of the crystal and to update estimates of the spatial distortions of the detector.

It is hoped that a summary of data collected on our system can be presented.