16.7-1 X-RAY DIFFRACTOMETER FOR STRUCTURE INVESTIGATION OF SURFACES. By D. Dornisch, F. Kretschmar, H. Schulz, U. Tangermann, D. Wolf. (Inst. f. Kristallographie, Universität München, SFB 128).

The application of X-rays offers new possibilities for the investigation of surfaces structures with large superstructure cells and complicated reconstructions. Up to now many such atomic rearrangements cannot be determined by low energy electron diffraction (LEED).

In the first successful surface structure studies using X-rays the crystal was prepared in a separate ultra high vacuum (UHV) chamber and afterwards moved to the diffractometer within a transfer system [1,2]. This technique does not allow the simultaneous surface control by other methods during the X-ray data collection, which needs a relatively long time due to weak surface signals.

Therefore we have developed a combined chamber allowing both, X-ray diffraction and in-situ surface control by LEED, and other surface sensitive methods. Further common techniques for crystal surface preparation are available including AES, TDS, gas adsorption and temperature treatments from 150K up to 2800K.

The diffraction geometry differs from the standard X-ray geometry in the way that the crystal is rotatable around two axes, the surface normal and an axis within the surface plane. In the zero position the X-ray primary beam (generated by an 18 - KW rotating anode) falls under glancing incidence on the vertically oriented surface. For use with synchrotron radiation the specimen holder can be repositioned such that the crystal surface is horizontally oriented, if required. The counting tube can be rotated around two axes, within the horizontal plane by 170° and perpendicular to it by ±40°, extending the accessible range in the reciprocal lattice from zero to high values of momentum transfer normal to the surface along reciprocal lattice rods.

The mechanics is characterized by the rotation of the crystal inside the fixed UHV - chamber. Accuracies are 0.002° for both the polar and azimuthal angles within a range of 0 - 130° and 0 -70°, respectively. The high angular precision of the azimutal rotation is achieved by mounting one bearing outside the differentially pumped flange. The bearings for the other rotation lie inside the UHV, but have comparable precision. The crystal can be adjusted into its zero position by a rotation around the primary beam within ±1.3° in steps of 0.0004° and xyz-translations in steps of μm. Without losing the orientation the crystal is moved within a minute from the LEED and preparation site into a beryllium cylinder for X-ray measurements. All movements, important experimental parameters and the data collection are controlled by a personal computer (AT).

The W(001) surface will be used for the first structure investigation. This surface exhibits a $(\sqrt{2}\times\sqrt{2})$R45°-reconstruction below room temperature [3] and requires frequent surface preparation due to the high reactivity of the surface with Hydrogen from the residual gas. The structure is believed to include small variations of atomic positions normal to the surface. The number of data points measurable with the apparatus presented here is sufficient to achieve a high accuracy for structural parameters normal to the surface.

[1] Brennan P., Eisenberger P., Nucl. Instr. Meth. 222 (1984) 164 - 167.
[2] Fuoss P.H., Robinson I.K., Nucl. Instr. Meth. 222 (1984) 171 - 176.
[3] Debe M.K., King D.A., March F.S., Surf. Sci. 68 (1977) 437.

16.7-2 *APOCALYPSE* NOW: UPDATE ON AUTOMATED PROTEIN CRYSTALLIZATION USING THE NEW ACA VAPOR DIFFUSION PLATE. By Noel D. Jones, John K. Swartzendruber, Jack B. Deeter, Paul W. Landis and David K. Clawson. Lilly Research Laboratories, Eli Lilly and Company, Indianapolis, IN 46285 USA.

APOCALYPSE is a fully automated protein crystallization system (N. D. Jones, *et al.*, abstract H4, ACA meeting, Austin, Texas, March, 1987) for large scale screening of conditions for protein crystallization with little operator intervention. Development of the hardware and software for phase one, GENESIS, is essentially complete. The hardware for REVELATIONS, the second phase, is in place and imaging software is being written.

GENESIS uses robotics automation to set up approximately 30 sandwiched drop crystallizations per hour in the new vapor diffusion protein crystallization plates (N. D. Jones and K. B. Ward, abstract PA26, ACA meeting, Hamilton, Ontario, June, 1986) which are available through the American Crystallographic Association. The robot and a bank of automated syringes which accurately dispense, dilute and mix solutions of protein, buffer, precipitants, salts and other components, are under computer control. The menu driven, user friendly software calculates the concentrations of the constituents for each well on the barcoded plates and records the information in a permanent data base.

REVELATIONS, when fully operational, will use robotics and a vision system to examine 100 to 200 protein drops per hour for the presence of crystals. The images from a high resolution color video inspection station will be digitized, analyzed and those selected stored on video disk for later retrieval. We hope to use optical density and edge detection to distinguish among clear drops, those with precipitate and those containing possible crystals.

16.7-3 AUTOMATED PROCEDURE FOR HANDLING ANOMALOUS DISPERSION CORRECTIONS IN THE GENERAL CASE FOR WHITE OR CHARACTERISTIC RADIATION DATA. By M.J. Mendelssohn and H.J. Milledge, Crystallography Unit, Dept. of Geological Sciences, University College London, Gower Street, London WC1E 6BT, UK.

Although it has long been recognised that anomalous dispersion effects in the vicinity of absorption edges can be useful both for absolute configuration determination and, in favourable cases, for phase determination, it is not always realised that quite substantial effects may occur in cases where they had been thought to be negligible, and so we have devised a procedure which, though primarily intended for use with Laue patterns, can also be used to optimise the selection of reflections measured with characteristic radiation if it is desired either to utilise or indeed to avoid anomalous dispersion effects.
As part of the evolution of "expert systems" for data collection intended to minimise the amount of data required to achieve defined crystallographic objectives in the most cost-effective manner (Milledge et al., Structure and Statistics in Crystallography, Ed. A.J.C. Wilson, Adenine Press, 1985), the development of an automatic procedure for selecting the optimum crystal orientation for observing anomalous dispersion effects on Laue films is an obvious requirement, and the necessary routines have been incorporated into our existing suite of programs.