**Laboratory Notes**


**A simple automatic cassette for X-ray synchrotron topography**

Use of synchrotron radiation for X-ray topography has led to an enormous reduction in exposure times and the possibility of rapid step by step experiments to study such phenomena as dislocation dynamics or magnetic domain wall motion. The rapidly expanding field has been reviewed recently by Tanner (1977). However, even though the exposure time may be as little as a few seconds (and with the advent of X-rays from storage rings this will be reduced even further), the time required to change manually the X-ray plate, clear the experimental area of personnel and open the shutter is typically two minutes. In short, experiments are severely limited if plate changing is not automated.

To promote rapid insertion and withdrawal of the Ilford L4 25 μm nuclear emulsion plates into and out of the diffracted beam a slide projector has been used in reverse as follows. The focusing lens was removed and a hollow brass cylinder was inserted in its place to eliminate X-ray damage to the plastic casing. Each X-ray plate (2 x 2") was mounted in a horizontally mounted circular slide magazine. The aluminium frame was necessary to prevent the thin plates from jamming. The loaded projector was then encased in a light-tight black plastic bag and positioned in the experimental area so that an X-ray plate would receive a particular X-ray reflexion when moved into the normal slide projection position. The quartz halogen projection lamp was removed and a lead screen was placed in front of the projector to cover all but the brass tube to avoid fogging of the stacked plates.

The experiment could then be completely automated and controlled remotely from outside the experimental area. Multiple exposures were affected simply by advancing the slide magazine by one. The latter operation took approximately 1½ s, meaning that the exposure time for the plate.

Because of chemical reaction between the aluminium, nuclear emulsion and developer the plates needed to be removed from the frames prior to development. In future, plastic frames will be used, and the plates developed in their frames.

The device has been used on the synchrotron radiation facility at Daresbury Laboratory and the SRC are thanked for provision of this facility.

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**Reference**


**Crystal orientation where the calculated adjustment exceeds the allowed movement on one arc**

During the process of crystal alignment, when the calculated adjustment required exceeds the allowed movement on one arc, correction may still be made by rotating about the mounting axis of the goniometer head. A method is described of calculating the adjustments required.

Nomenclature and sign convention are as shown in Fig. 1. The unit vector, \( \mathbf{u} \), lies along the crystal axis and its coordinates are described by its projection on the xy plane. Consider \( \mathbf{u} \) coincident with the z axis when the arcs are at their calculated angles \( (\varphi_T \text{ and } \varphi_B) \) for alignment. Rotation of the top arc to 0° followed by similar rotation of the bottom arc gives \( x = \sin (\varphi_T) \) and \( y = \cos (\varphi_T) \sin (\varphi_B) \), the signs being negative because rotation is to rather than from the origin. The vector \( \mathbf{u} \) is constrained under a rotation \( \omega \) about the z axis to a circle in the xy plane. If \( \varphi_T \text{ and } \varphi_B \) are the maximum allowed adjustments on the arcs then the region accessible is given by

\[
x = \sin \varphi_T \text{ and } y = (1 - x^2)^{1/2} \sin \varphi_B,
\]

the second condition limiting movement to within an ellipse of semi-axes unity along x and \( \sin \varphi_B \) along y. The forbidden region is shaded in Fig. 2. Adjustment is possible if and only if the circle described by the rotation of \( \mathbf{u} \) enters the allowed region.

The points of maximum adjustment are the points in the allowed region most distant from the origin, i.e. \( x = \sin \varphi_T \text{ max}, \)

Fig. 1. The coordinate system used to describe a unit vector along the crystal axis. The sign convention used is 'left is positive' on the top arc while 'right is positive' on the bottom arc since these rotations will move the vector toward the positive axes. (Adapted from an arc diagram supplied by Stoe & Co.)

\[
\omega
\]

Fig. 2. The permitted region (x horizontal, y vertical) corresponding to \( \varphi_T \text{ max} = \varphi_B \text{ max} = 15^\circ \). The vector for \( \varphi_T = +17^\circ \text{(L)} \) and \( \varphi_B = +2^\circ \text{(R)} \) is shown by the dashed line. Correction can be achieved by rotation to the position of any of the four solid arrows, the nearest requiring a movement of 37.5° (anticlockwise) and arc settings of \( \varphi_T = +12.2^\circ \text{ and } \varphi_B = +12.1^\circ \).
Laboratory Notes

Two modifications of commercial units available for the automation of X-ray diffractometers

Philips diffractometers can be upgraded to automatic operation by modest additional investment if the automatic sample changer and its control unit are added to the basic equipment. However, it was found that this set-up can be further improved in two respects. (1) It is desirable that any sample out of the 35 loaded into the cartridge should be directly selectable for the next measurement. With the original control one cannot avoid having to begin at the first sample and then proceeding one-by-one in increasing numerical order. (2) Synchronous start of recording at a thick line of the chart and of scanning from an integer 2θ value should be made possible in order to achieve easy evaluation of 2θ values from the powder diffractograms. The following simple and inexpensive circuits were introduced to provide solutions to these problems.

Free specimen selection was obtained by applying to the inputs of a comparator both the voltage indicating the actual sample number in the original PW1170 unit and that proportional to the desired sample. The latter was supplied by a supplementary source and divider. The difference signal of the comparator drives, through an amplifier, the positioning motor until the desired sample is set into the loading position. Then, the normal sequence of operations is continued. The number of the sample selected for the next run can be preadjusted by two switches (corresponding to the two digits). Synchronization of angular markings to the scale of the chart requires a special transducer. A brass disk—mounted on the axis of the paper-driving drum (Fig. 1)—with insulator inserts around its periphery separated by distances corresponding to the periodicity of the thick lines present on the recording paper, and a sharply tipped contact spring sliding on this surface yield an adequate source of impulses for synchronization. Full match between angular marks and thick lines is achieved by means of a circuit sensing the signals coming from the angular limiting switches of the diffractometer and the above mentioned transducer, and controlling the driving mechanism of the goniometer and that of the chart recorder respectively.

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Marking of spherical crystals for identical re-mounting

Dots of Indian ink can be used to mark directions on a spherical crystal so that it can be re-mounted in exactly the same orientation as it was mounted in a previous experiment. Examples of need for this occur in studies of the effects on X-ray intensities of various treatments of a particular sample which must or may be demounted between X-ray examinations. Further, the crystal symmetry may be changed by the treatment, and reflections which were equivalent may then differ. This is the case for quartz crystals after an irradiation with fast neutrons of about $3 \times 10^{18}$ n cm$^{-2}$.

In general, two marks, one along the setting axis and one perpendicular to it, are enough to permit one to recover the orientation even if the crystals has become separated, whether by design or accident, from the fiber or pin on which it was mounted for X-ray study.

The directions can conveniently be indicated with small Indian-ink dots applied with the help of a micro-manipulator and a binocular microscope. A fine cotton thread is attached to the micro-manipulator and its tip, previously dipped into Indian-ink, is brought into contact with the crystal under the microscope. After some practice, dots of about a few hundredths of a millimeter can be made without excessive difficulty. Different colors may be used to distinguish the two different directions, but colors tend to disappear with irradiation; it is perhaps preferable to use two adjacent Indian-ink dots to mark one of the axes.

If very precise marking of the directions is not needed one can make all the manipulations on a table with the crystal mounted on the goniometer head. If better precision is needed, then the manipulations can be done with the crystal mounted on the diffractometer, the axes to be marked being brought successively to the position of the Indian-ink bearing cotton fiber.

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Stereoviews and morphological data on micro crystals

With existing crystallographic computing programs it is possible to draw stereoviews of a crystal and to calculate its interfacial angles with three-dimensional coordinates of the vertices defining the crystal. These coordinate data can be obtained from a series of two-dimensional views with either an optical or an electron microscope.

A clean crystal of fructose with well developed faces (15 μm) was attached to