SYNCHROTRON X-RAY SECTION TOPOGRAPHY (SST)

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The X-ray topographic technique most commonly practised with non-monochromatised, white synchrotron radiation employs a broad incident beam to flood most or all of the area of the specimen crystal that is of concern for the investigation in progress. The Bragg diffracted beam may be recorded as a reflection from the crystal surface: this is clearly the appropriate experimental mode for imaging travelling surface acoustic waves, for example. In a more commonly used mode, the Bragg diffracted beam transmitted through the crystal is recorded. The transmission mode is most often applied to plate-shaped specimens which are not very highly X-ray absorbing. Its power resides in its ability to reveal non-destructively the population of lattice defects (e.g. dislocations) within the interior of the crystal. An illustration of this method, a study of a polished slice (about 0.2 mm thick) of natural beryl, may be cited. Sometimes, however, the location of a particular defect buried within the crystal needs to be pinpointed, and maybe also have its diffraction contrast investigated in detail. To achieve these objectives, one reverts to the section topograph technique, the first of the high-resolution X-ray topographic methods to be developed, and that with which the X-ray diffraction contrast images of individual dislocations were first recorded.

Figure 1 recalls the diffraction geometry of the section topograph. It is a plan view of the plane of incident and diffracted rays. In this technique the width of the beam entering the crystal is limited by the slit S to form a narrow ribbon, extending normal to the plane of incidence (i.e. parallel to the goniometer axis) and covering the height range of interest in the specimen. Traces of the X-ray entrance and exit surfaces of the specimen are shown passing through O and AB, respectively. (For simplicity these traces are drawn parallel to each other: in practice the specimen can have an irregular shape.) Traces of the Bragg planes
active are indicated by the set of parallel lines C. X-rays entering at O and diffracted by planes in the orientation of C fan out within the triangle AOB enclosed between the direct beam and diffracted beam directions, OA and OB, and whose opening angle OAB is $2\theta_B$, twice the Bragg angle. In a reasonably perfect crystal the X-rays undergo multiple scattering within this 'energy-flow triangle' AOB. Hence in addition to the diffracted beam leaving the crystal between A and B (and falling upon the photographic plate P between $A'$ and $B'$) there also exists a wide forward-scattered beam lying between the rays $AA''$ and $BB''$. (Generally, this forward-scattered beam is not recorded, though it is sometimes of interest to do so: e.g. in the case of forward-scattered ($K_o$-beam) and Bragg-scattered ($K_g$-beam) stacking fault image patterns. The $K_o$-beam and $K_g$-beam patterns are analogous to the 'bright-field' and 'dark-field' images familiar in transmission electron microscopy). Two essential ingredients of the section topograph image of a nearly perfect crystal are shown schematically in Fig. 1. Firstly, suppose the crystal is defect-free except for a single dislocation threading through it along the path $D_1D_2$. One must visualise $D_1D_2$ as lying along the axis of a filament of 'bad' crystal within which the lattice misorientation exceeds the angular range of Bragg reflection by the perfect matrix. In comparison with perfect crystal, this filament 'accepts' for Bragg reflection radiation covering a wider $\Delta\theta$ range, in the case of monochromatic radiation, or a wider $\Delta\lambda$ range, in the case of collimated white radiation, and (provided the experiment is not done under conditions when 'anomalous transmission' greatly enhances the intensity from perfect matrix) a strong 'kinematically diffracted' beam issues from the point K where OA cuts $D_1D_2$. This beam falls on the plate P to produce a dot of blackening at $K'$. Thus the locus of $D_1D_2$ (or of any other defect producing a 'kinematic' image) can be mapped by taking a sequence of section topographs with OA cutting the specimen at precisely known locations. The second essential ingredient of the section topograph image is the Pendellösung fringe pattern. In the course of the multiple scattering referred to above, the X-ray energy-flow within AOB
swops back and forth between the $K_o$ and $K_g$ beams: this oscillation constitutes the Pendellösung phenomenon. The dynamical diffraction theory appropriate to conventional X-ray source conditions (the 'spherical wave' theory) shows that loci of maxima of $K_g$-direction energy flow in the crystal lie on hyperbolae having OA and OB as asymptotes, as sketched. (Maxima of $K_o$-direction flux lie on similar hyperbolae, interleaved between those sketched.) The actual spatial periodicity of intersections of each set of hyperbolae with the Bragg planes (i.e. the Pendellösung period) depends upon wavelength, structure factor and $\theta_B$ and is typically in the range 20 μm to 200 μm. The Pendellösung fringe pattern recorded on P is determined by the geometry of intersection of the crystal's X-ray exit surface AB and the hyperbolae within AOB: diffracted rays corresponding to Pendellösung fringe maxima follow the interrupted lines in Fig. 1. (Regarding details of the theory and applications of the section topograph, the review by Authier is informative.)

From Fig. 1 it can be seen that with crystals whose thickness is around 1 mm, say, and with typical Bragg angles such that $2\theta_B \sim 45^\circ$, a 10 μm wide incident beam passed by S will be spread out into a diffracted beam whose width $A'B'$ is in the region of 1 mm. As a consequence of this beam expansion, and the resulting decrease in photon flux per unit area reaching the recording medium, the taking of high-resolution section topographs with conventional X-ray sources is a slow procedure. On the other hand, use of the SRS reduces exposure times by two orders of magnitude. Thus synchrotron X-ray section topography (SST) allows section topographs of good quality and high resolution to be obtained with exposure durations no longer than the photographic plate processing time. Such a working schedule offers an experimental throughput fast enough for any non-dynamic studies of defects. However, the twin demands for (a) precise control of the position of the X-ray entry point O on the crystal and (b) maximum productive use of SRS beam time prompted development of a self-contained device which allows setting-up of the experiment on a conventional X-ray topographic camera followed
by a speedy and reproducible transfer to the White Radiation Camera (WRC) at Daresbury. A key feature in the success of the device is the employment of optical alignment techniques, as detailed below.

Figure 2 shows highly schematically the X-ray and optical beam paths, and the several angular adjusting facilities possessed by the apparatus in its current state of development. As Fig. 3 shows, it now constitutes a 'mini-camera' designed for attaching to the specimen-carrying spindle of the WRC. The assembly in Fig. 3 can be divided at the point shown schematically at X in Fig. 2: unscrewing the fluted ring seen below A in Fig. 3 allows detachment of the specimen-carrying goniometer head and its attached members which may then be transferred to the conventional X-ray topographic camera. Components of Fig. 2 visible in Fig. 3 are similarly lettered. In Fig. 2, solid lines represent the X-ray paths: from the tangent point T, through a 'coarse' beam-limiting slit Q, then through the fine slit $S_1$ (equivalent to S in Fig. 1), and through the specimen crystal C to a removable beam-stop Z. The diffracted beam from C falls on the photographic plate P mounted on the rotatable arm D. (The plate-holder is quickly removable from its support by undoing knurled nuts which may be seen in the upper left in Fig. 3.) There are also two light sources (not shown in Fig. 3). One is upstream of Q, shown in and out of the X-ray beam path at I and I' respectively. This light simulates the X-ray source. The second light source is in the autocollimator W which is inserted in the tail bearing of the WRC. W may also be used as a telescope for viewing C, $S_1$, Q and I by addition of a long-focus lens (shown at L and L' in and out of the light beam path, respectively). Proceeding outwards from the central axis, the parts shown in Fig. 2 are as follows. The crystal C is mounted on a goniometer head H to which is fixed a collar A carrying an angular scale 'a'. Riding on A is a rotatable sleeve B which carries (i) a removable fine X-ray slit $S_{1'}$ (ii) in position diametrically opposed to $S_1$, optionally either another removable slit $S_2$ or (as shown in ref. 7) a removable mirror, (iii) a fixed mirror M which is viewed by the autocollimator W and whose
normal is adjusted to be parallel to the diameter $S_1S_2$, and (iv) a micrometer tangent screw (see Fig. 3) for fine adjustment of the angle between A and B. (Two micrometers on brackets are available: they can be attached to whichever side of B is more convenient for the experiment in progress, or they can be dispensed with when finger-tip adjustment of B relative to A (i.e. giving settings to about $1/3^\circ$) is adequate.) The plate-carrying arm D is rotatable relative to the mount E which carries the goniometer head H. The angular position of D relative to E is read on a scale 'e' fixed to E and viewed through a slot cut in D. (No fine adjustment for this angle is needed or provided.) For fine angular adjustment of E relative to the base F (i.e. the angular setting represented by the scale 'f') a lockable radius arm and tangent screw are fitted, as seen in the lower left of Fig. 3. Finally, the angular setting of the whole assembly, after attachment to the specimen axis of the WRC, is determined by the relevant scale 'g' belonging to the WRC (the latter being represented by G in Fig. 1). This last-mentioned setting is fixed at the beginning of the X-ray experiments, all subsequent angular settings being made relative to an optically defined zero angle determined by centring the light beam reflected from M on the zero of the auto-collimator scale. Assume that the angle between the normal to M and the Bragg plane normal in the specimen is known (and if the specimen has natural reflecting facets, or optically polished surfaces in known orientation, this angle may be determined in situ using W plus the rotational freedoms provided to serve as an optical goniometer). Then, all the adjustments needed for a series of section topographs in which changes of Bragg plane and/or wavelength are made can be done using the coarse and fine angular adjustments provided on the assembly shown in Fig. 3. The adjustment routine is as follows. Starting with $S_1S_2$ aligned with the normal to M (as earlier mentioned), the desired crystal orientation change $\omega$ is set using 'f'. The setting on 'a' is then changed by $-\omega$ to re-align $S_1S_2$ with the X-ray beam axis (a re-adjustment easily checked using M and W). The desired $2\theta_B$ setting for D is set on 'e' after subtracting the reading on 'f'.
The use of the auto-collimator's own angular scale (one minute of arc divisions) is very helpful in checking that in the rush of experiments no error in sign or magnitude of small changes in 'a' and 'f' is perpetrated. Moreover, when $\theta_B$ is relatively high (say $20^\circ$ or more), the easiest way to make small shifts of the position $O$ of X-ray beam impingment on the crystal (shifts in the range 10 to 200 $\mu$m, say) is to rotate $B$ by the required fraction (generally small) of a degree. Unless working close to an absorption edge, the changes in diffraction parameters resulting from the consequent small change in $\lambda$ are insignificant.

The SST reproduced in part in Fig. 4 is a (111) reflection given by a 1 mm thick parallel-sided polished (001) plate of natural diamond. The left and right hand margins of Fig. 4 correspond to $A'$ and $B'$ of Fig. 1, respectively, the actual distance $A'B'$ being 0.75 mm. The following features are noteworthy. Firstly, the wavelength chosen, 0.1 nm, proves extremely useful for X-ray topography: at this wavelength the SRS has no competitor in the way of characteristic radiation sources. Secondly, this diamond is unusually perfect: no dislocation images are cut by this section of the crystal, and the regularity of the Pendellösung fringe pattern testifies to a rare freedom from long-range strains. Lastly, the pattern is that expected from a 'spherical wave' source (i.e. one giving rise to hyperbolae filling $AOB$, as described above). But the source was 80 m distant, and should behave more nearly as a 'plane wave' source! This topic we hope to discuss in a future Bulletin.