Applications of ‘limited projection topographs’ and ‘direct beam topographs’ in diffraction topography

A. R. LANG

H. H. Wills Physics Laboratory, University of Bristol

MS. received 2nd August 1963

The method of projection topographs can be modified so that images are recorded only from imperfections which lie within a chosen range of depths in the crystal specimen. This technique of ‘limited projection topographs’ allows easier study of internal imperfections by eliminating images of surface damage. It can also be applied in studying imperfections just below a crystal surface, as in correlating dislocation outcrops with surface features. Another modification uses multiply scattered radiation leaving the crystal in the direction of the direct beam to produce ‘direct beam topographs’ which have different contrast characteristics from the usual diffracted beam topographs.

1. Introduction

The method of x-ray ‘projection topographs’ (Lang 1959) was designed to present an easily interpretable overall picture of the distribution of lattice imperfections within a crystal. The image recorded on the topograph is a projection of the entire crystal volume scanned by the incident x-ray beam, the direction of projection being that of the diffracted rays of the chosen Bragg reflection. The area of specimen scanned during an exposure is limited only by the maximum available height of incident beam and by the maximum range of the linear traversing mechanism carrying the specimen. Specimen thickness is obviously limited by absorption of the x-rays, but with light elements and radiations such as MoKα, AgKα and W Kα, specimens up to several millimetres thick can be used. However, with thicker specimens it may often happen that one wishes to concentrate study only on a certain range of depths within the crystal, and to do this without mechanically thinning the specimen. This need arises when the specimen surfaces are unrepresentative of the bulk specimen to an appreciable depth, or when the specimen has surface damage which it is impossible, or not desired, to remove. Also, since imperfections lying throughout the whole depth of crystal have their images superimposed on the projection topographs it follows that the capacity for resolving individual defects (such as dislocations) decreases as the specimen thickness is increased. If the specimen could be examined layer by layer in turn, less crowded patterns of dislocations would be obtained. These requirements may be met by use of the ‘limited projection topograph’ described in §2, an arrangement which usefully enlarges the field of application of x-ray topographic methods.

In the usual experimental arrangement for taking projection topographs the beam incident upon the specimen is not collimated by reflection at a monochromator crystal; in fact, the use of an incident beam whose angular divergence is wider than the natural angular width of reflection of a perfect region of the specimen contributes to the production of high diffraction contrast at imperfections, when the specimen is lightly absorbing. Occasionally this high contrast is not required, and is indeed undesirable when it comes from locally intensely deformed regions such as those surrounding cracks, inclusions and surface scratches. In such cases a more strictly collimated incident beam is wanted in order to prevent highly misoriented regions from giving their intense Bragg reflections. Strict collimation may be achieved by use of the double or triple crystal spectrometer arrangements, but at the cost of considerable complication of the instrumentation and the necessary adjustments, and of great sensitivity to thermal and mechanical instability. One very simple way of achieving the effects of strict collimation is to use the specimen crystal itself as its own ‘crystal collimator’. In this technique the topograph image is produced by radiation which has undergone multiple reflection in the specimen crystal and which leaves the crystal in the direction of the incident beam. Such a diffraction arrangement may be called ‘direct beam projection topography’ and will be more fully described in §3.

2. Limited projection topographs

Figure 1 shows in plan several variants of the experimental arrangements for taking projection topographs. The incident beam, which has the form of a narrow ribbon perpendicular to the plane of the drawing, cuts the specimen crystal C along OA. Bragg reflection occurs at planes...
parallel to OP, giving rise to diffracted rays such as OB. The photographic emulsion is placed at F to record diffracted beam topographs or at F' to record direct beam topographs. (If a thin emulsion is used, so that oblique incidence does not lead to significant loss of resolution, both the diffracted beam topograph and the direct beam topograph can be received on the same plate, set perpendicular to OP.) During the course of exposure the plate and the specimen are together translated backwards and forwards parallel to the double-headed arrow in order that the entire specimen volume of interest is cut by the direct beam OA. In the standard arrangement for taking projection topographs the aperture in the stationary screen between C and F is set like S1. For diffracted beam topographs this screen does, of course, extend sufficiently on either side of the diffracted beam to intercept the direct beam and any unwanted other x-ray reflections. Diffracted rays are received on F from the whole depth of the crystal: rays directly diffracted from the point O on the entrance surface travel along BR' and those directly diffracted from the point A on the exit surface travel along AR. The aperture required in S1 is a minimum when the direction of specimen traverse is made parallel with the entrance and exit surfaces (if they are parallel to each other) or with the mean plane of the specimen (if it tapers). The specimen mean plane is usually set normal to the plane of the incident and diffracted beams.

Figure 2 is a diffracted beam projection topograph of a natural diamond octahedron. The crystal surfaces are much damaged by ring-cracks and scratches and the topograph is covered by their intense diffraction images. The strong diffracted intensity produced by the extra damage at edges and corners outlines the projection of the octahedron. Stereo-pairs of projection topographs show that the strong spot seen to the left of the topograph centre comes from an inclusion within the stone, but a cursory examination of the topographs suggests that there are no other internal imperfections. As is shown by its symmetry, the topograph of figure 2 was taken with a pair of octahedral faces set normal to the plane of the incident and diffracted beams. Hence these faces correspond to the x-ray entrance and exit surfaces of the specimen C drawn in figure 1. Now let the aperture in S1 be replaced by the reduced aperture S2. The only rays directly diffracted from the incident beam and admitted by S2 are those originating within the volume between the planes XX' and YY'. Hence, over most of the topograph, no directly diffracted rays coming from the damaged faces of the crystal can reach F. On the resulting 'limited projection topograph', figure 3, almost the same effect is achieved as if the damaged entrance and exit surfaces had been physically removed. Defects within the interior of the crystal are more easily detected since the obscuring background of surface damage has been eliminated. In particular, about half a dozen dislocations, radiating from a small inclusion situated below the major inclusion, are now seen clearly. In the full projection topograph (figure 2) they are largely hidden by surface damage and can be recognized only by careful examination.

Another application of the limited projection topograph is shown in figures 4(a) and 4(b). The specimen is a plate of silicon, about 1-2 mm thick, cut roughly parallel to one of the slip-planes, which are of the type {111}. There is no surface damage problem here since the crystal can readily be etched. Dislocations lie in the slip-planes parallel to the specimen, at various levels within it, and also to some extent on the other {111}-type slip-planes steeply inclined to the specimen plane. Since the specimen is relatively thick, and images of dislocations in silicon formed with AgKα radiation are rather broad, the full projection topograph, figure 4(a), becomes confused at a relatively low dislocation density. Some clarification of the dislocation distribution can be gained by examining the specimen layer by layer. Figure 4(b) is a limited projection topograph of the same region as figure 4(a), taken with the same Bragg reflection but with the diffracted-beam aperture in the position S3 of figure 1. This setting allows directly diffracted rays to reach F from about one-third of the thickness of the crystal. Hence those dislocation loops lying within this fraction of the specimen thickness, from the exit surface down to the plane ZZ', may be clearly identified.

The fraction of the full diffracted-beam width RR' which is allowed to reach F may be made as small as desired, and in practice this selection is facilitated by a micrometer.
adjustment. However, the necessarily finite width of the
direct beam OA (for which 12 to 15 \( \mu m \) is probably the
practicable minimum value) imposes some uncertainty on
the definition of location of the planes XX', YY' and ZZ'.
One obvious application of the S3 arrangement, in which
the exit surface and a small depth below it is selected for examina-
tion, lies in the correlation of dislocation outcrops with
visible surface features on the crystal. An example of such
work is the finding that pyramidal trigons on diamond
octahedral faces coincide with dislocation outcrops (Lang
1964). The limited projection topograph technique may find
useful application in studies of epitaxial growth on relatively
thick substrates.

![topograph](image)

**Figure 4.** (a) Projection topograph of silicon plate, field
width 3 mm, 220-type reflection, AgK\( \alpha \). (b) Limited pro-
jection topograph corresponding to figure 4(a), showing
one-third of full specimen depth.

The greatest contribution to the high diffraction contrast
manifested by imperfect regions (under conditions of rela-
tively low x-ray absorption) comes from the 'direct diffraction
image' formed when the imperfection is cut by the direct
beam OA. As has been shown, any of these diffracted rays
can be prevented from reaching the emulsion, as need arises.
However, faint images of imperfections can still be produced
by other mechanisms. For example, near the centre of the
full projection topograph, figure 2, the direct images of two
bad scratches are seen. In figure 3 these direct images are
eliminated but the scratches are still faintly visible by their
effect on other diffracted rays passing through them. Also,
both experiment and theory show that rays participating in
the process of multiple coherent scattering within a relatively
perfect crystal fan out in all directions included between the
primary beam and diffracted beam direction (see, for example,
von Laue (1960)). Such rays, originating at or passing
through an imperfection slight enough only to disturb but
not destroy the multiple scattering process, produce a weak
diffuse 'dynamical diffraction image' of the imperfection.
When the imperfection is near O (figure 1) its dynamical
image spreads out over most of the base AB of the triangle OAB,
and is hence difficult to eliminate by any scaling of the
diffracted-beam slit. The diffuseness of the dynamical image
in this case makes it unimportant. It is possible, however,
to use the technique of limited projection topographs to
enhance the contrast of the dynamical images of slight
imperfections in nearly perfect crystals. In the fan of rays
included between the direct and diffracted beam directions
it is the central ones, flowing parallel to the Bragg planes
OP, which are the most sensitive to slight crystal distortions.
Hence a slit-setting such as S2, which admits rays coming
from P but intercepts those coming from the margins of the
diffracted beam, increases sensitivity in the detection of very
weak lattice distortions. It has been used successfully to
enhance the contrast of projection topograph images of
oxygen bands in crucible-grown silicon crystals.

3. Direct beam topographs

If the range of misorientations of the crystal is small then
multiple scattering of x-rays will occur within the triangle
AOB whenever the incident beam OA covers the angular
range of Bragg reflection of planes parallel to OP. A natural
result of such multiple scattering is that the direct beam is
spread out laterally from AT to BT. The expected intensity
profiles across the diffracted beam RR' and the direct beam
TT' can be calculated from the dynamical theory of x-ray
diffraction when the crystal is sufficiently perfect for the
multiple scattering to occur coherently (Kato 1960). The
intensity across TT' falls off rapidly from T to T' in cases
of low or moderate x-ray absorption, i.e. when the product
of linear absorption coefficient and crystal thickness does
not exceed a value of about 4. However, if a screen is placed
to cut into the edge of the direct beam, as S4, figure 1, just
far enough to intercept radiation in the incident beam which
has not undergone Bragg reflection, but not enough to
intercept an appreciable fraction of the multiply scattered
rays reaching TT', then projection topographs can be
obtained with the remainder of the direct beam comparable
in intensity with the usual diffracted beam topographs. In
simple terms, the essential difference between the 'direct
beam topographs' thus obtained and diffracted beam topo-
graphs is that the images in the former are produced by
rays which have undergone at least two reflections in the
crystal. Some of the conditions of the double-crystal
spectrometer arrangement are thereby simulated. It follows
that in direct beam topographs there is a great reduction in
the intensity of the direct images of imperfections, for all
parts of the specimen. The sensitivity to small lattice
distortions is not reduced, for perturbations of the sensitive
rays, which flow parallel to OP, affect the direct and dif-
fracted beams equally, but in opposite sense.

Figure 5(a) is a diffracted beam projection topograph of a
natural diamond octahedron. The usual damage to faces
and edges is evident. In addition, dislocations radiating
from the centre of the stone may be seen. The direct beam
projection topograph, taken at the same time, appears rather
different in shape due to the difference in projection directions,
the angle between direct beam and diffracted beam being
33° for MoK\( \alpha \) radiation and the 220 reflection. On figure
5(b) the strong images from misoriented regions surrounding surface damage are greatly reduced in intensity, for all surfaces. The dislocations can still be seen fairly clearly. Various mechanisms contribute to the diffraction contrast of dislocations on x-ray topographs. A comparison of diffracted beam and direct beam topographs throws some light on their working. In cases of low x-ray absorption and already mentioned, in a perfect crystal a fan of coherently scattered x-rays fills the whole of the triangle OAB. Thus, in the triangle, near A the energy flow is nearly parallel to OA, and near B it is nearly parallel to OB. Also, in the case of low x-ray absorption, the energy flow at A is much stronger than at B. It is to be expected, then, that a dislocation near the exit surface, acting as a strong x-ray reflector, will divert the dominant energy flow from the AT to the AR directions when situated near A, and from the BR to BT' directions when near B. This phenomenon has been well observed with silicon crystals and AgKα radiation. It is illustrated by the following experiment. With the screen setting S4, the direct beam topograph is formed predominantly by rays travelling close to AT on account of the above-mentioned distribution of energy flow across AB and the resulting fall-off in intensity, from T to T', of rays reaching F'. Thus the type of contrast on the direct beam topograph given by dislocations near the exit surface will be governed by their effects when near A. Since at this point they divert energy away from the direct beam direction they will appear as light lines on a darker background, i.e. with contrast opposite to that exhibited on diffracted beam topographs. Figure 6(a) is a diffracted beam topograph of an array of dislocations close to the exit surface in a plate of silicon. Figure 6(b) is the direct beam topograph of the same region of the crystal, with the screen set as S4. If the screen is pushed far into the direct beam, into the position S5, then, as expected, contrast of the same type as in figure 6(a) is regained, but the overall intensity of the pattern is weak.

Figure 6. (a) Projection topograph of silicon plate, field width 1.7 mm, 220-type reflection, AgKα. (b) Direct beam topograph of field of figure 6(a), 440-type reflection, AgKα.

Acknowledgments
The author gratefully acknowledges financial support from the Metallurgy Branch, United States Office of Naval Research, for that part of the work done at Harvard University, and from the Aeronautical Research Laboratory, Office of Aerospace Research, for support subsequently, through the European Office, Aerospace Research, United States Air Force.

References