Short Communications

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Observation of background contrast in convergent beam patterns.

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The relation of inelastic scattered intensity to the main elastically scattered pattern may be observed in convergent-beam patterns, with regard to incident and scattered directions and crystal thickness, and qualitatively compared with theory. The contribution of inelastic scattering to absorption and other measurements can be relatively easily estimated due to the sharp separation of the diffraction aperture, and the convergent-beam method has this advantage over electron-microscope imaging.

Introduction

Background contrast to convergent beam patterns has been observed by Kossel (1948) and Uyeda, Fukano & Ichinokawa (1954). More detailed observations show that this contrast is in some cases what it appears to be *viz.*, identical to the elastic contrast. Theory (*e.g.* Fujimoto & Kainuma, 1963; Howie, 1963) shows that this can arise from low-angle inelastic scattering. A requirement for this type of detailed contrast is that the scattering source effectively occupies the whole crystal depth, *i.e.* a crystal loss and not a core loss. With heavier atoms and/or different structures, core and thermal losses become relatively more important and a different contrast, described by classical Kikuchi line theory (Kainuma, 1955; Gjønnes, 1966) becomes evident. Even with the former type of loss some averaging of contrast occurs outside the angular range of incidence. This is shown by examples.

Examples

From MgO platelets thickness-dependent inelastic contrast can be observed with point patterns (Gjønnes & Watanabe, 1966). With convergent beam patterns under conditions of systematic interaction contrast appears as a detailed continuation of the elastic pattern fringe system into the background, and this applies to strong reflexions with appreciable excitation error, whose pattern is very thickness sensitive. This was tested by microphotometering the 200 distribution of MgO in a pattern with the 400 reflexion excited [Fig. 1(*a*)]. Strong and extensive contrast has been observed in semi-conductor crystals, *e.g.* Si (Gjønnes, 1970) and CdS. The CdS pattern is shown here (Fig. 1) and microphotometer curves (Fig. 2) taken across the pattern inside, and at different distances outside the elastic pattern show typical behaviour. Intensity profiles of the convergent beam pattern are followed in detail in the inelastic pattern, up to the envelope of 'accidental' lines. On crossing the envelope detail is lost; the Kikuchi lines continue but with reversed contrast. This means that contrast is preserved in multiple inelastic scatterings (because of the large angles involved), and is phase sensitive. Asymmetry with respect to the Kossel band from the break-down in Friedel's Law is followed in the background. This is true of the 00$\bar{2}$, 000, 002 beams. The convergent beam contrast of the weaker 004 beams is not followed, (note mis-match of lines at edge of 004 discs in Fig. 1) and the inelastic pattern in this region is *symmetric* with respect to the Kossel band. This scattering is indirect (from the strongly excited beams) and interference between different diffraction conditions occurs. Asymmetry of the diffraction from a non-centred structure is an

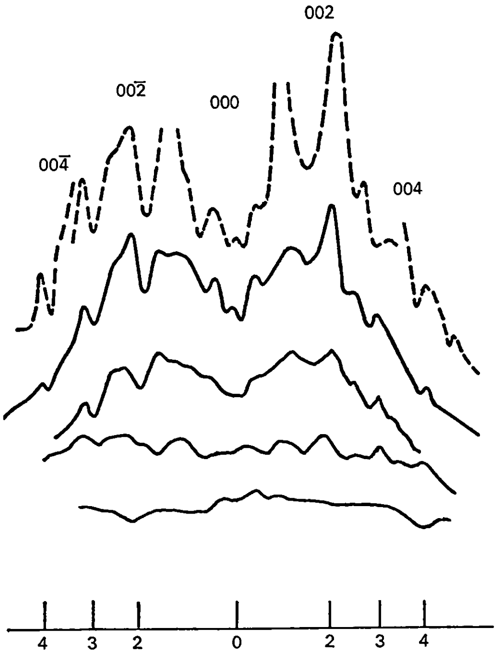
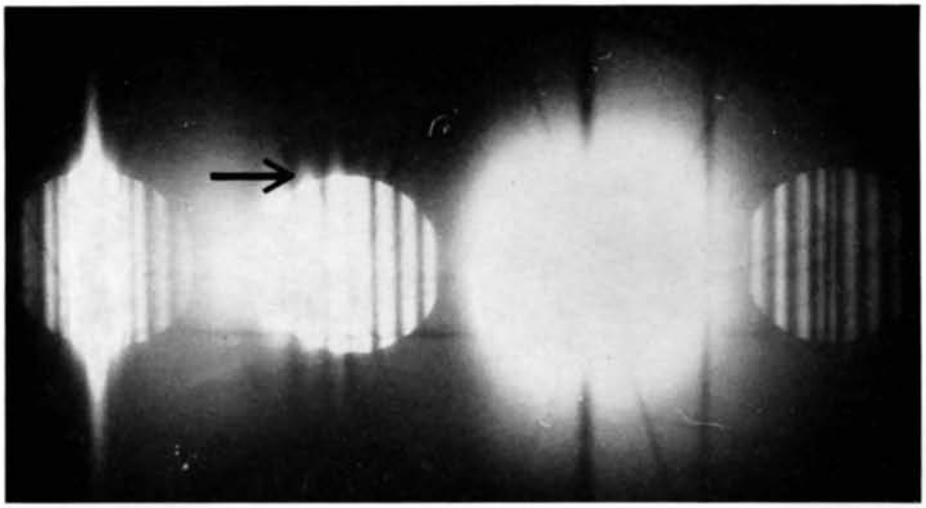
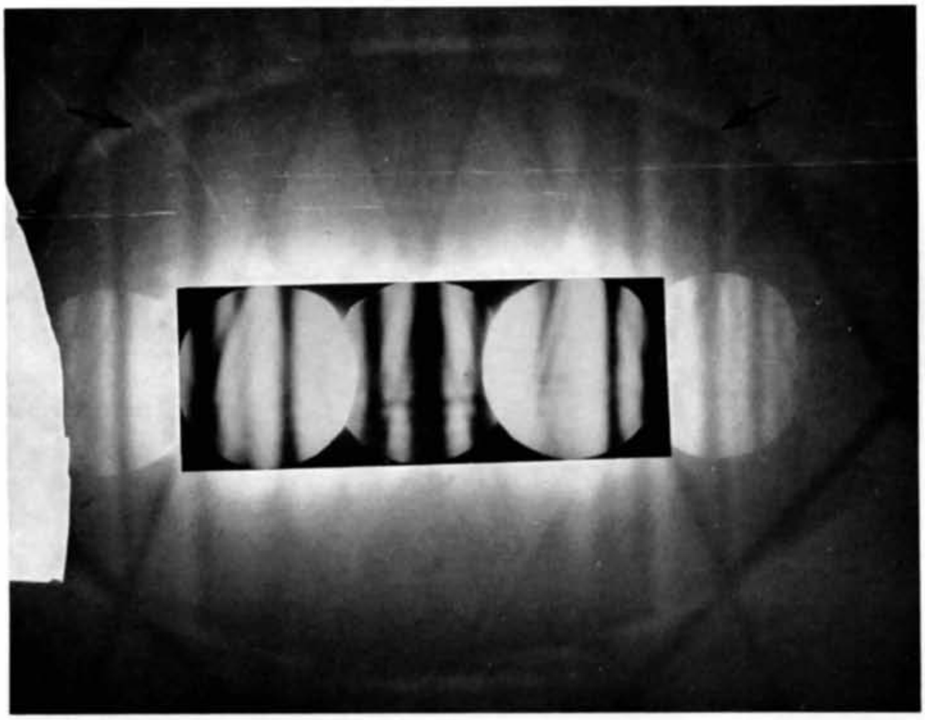


Fig. 2. Microphotometer scans from CdS pattern of Fig. 1(*b*). From top to bottom, convergent beam intensities (broken curve), inelastic pattern at increasing distance from the convergent beam apertures (two curves); the last curves are taken from just inside, and just outside, the (21$\bar{3}$1) envelope. In comparing these latter, note the lack of detail, and profile reversal, in the last curve. Horizontal scale corresponds to numbering of Kikuchi line pairs, in order from the centre of the band.

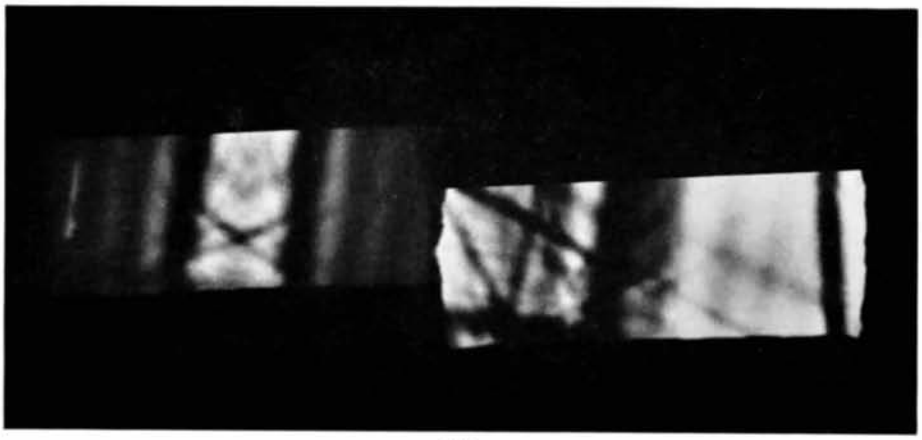


(*a*)



(*b*)

Fig. 1. Patterns showing background contrast. (*a*) MgO, showing 200 background fringes at high excitation error. (*b*) Pattern from $3\bar{2}\bar{5}0$ region of CdS. Central pattern is from a lighter exposure of the main beams. Accidental $(21\bar{3}1)$ envelope is indicated by arrows.



(*c*)

(*d*)

Fig. 1 (*cont*). (*c*) and (*d*) Under and over exposure of MoS_2 pattern with the 110 reflexion excited, at 100 kV and 50 kV respectively.

oscillating function of thickness, and sensitive to any averaging of phase. The problem of contrast arising from a strong structure factor, outside the angular range of incidence, is complicated and requires individual consideration, but the result is not necessarily identical to that predicted by classical Kikuchi* line theory which involves an integration over the Z coordinate (*i.e.* averaging of a different kind).

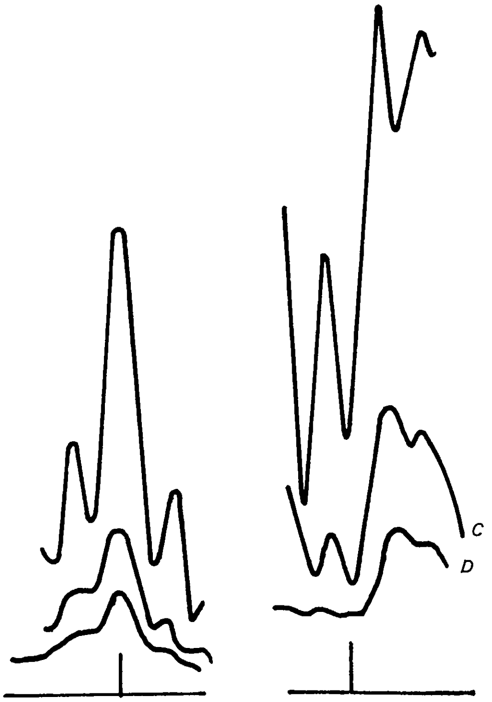


Fig. 3. Microphotometer curves from the MoS$_2$ pattern, from the 110 and 000 beams. From top to bottom the scans are, inside the convergent beam aperture, inelastic pattern close (C) and distant (D) from the aperture.

Patterns from MoS$_2$ [Fig. 1(*c*), (*d*)] with the 110 reflexion excited show, besides extended contrast as above, strong absorption asymmetries in the background. Asymmetry associated with the 110 background (Fig. 3) can come only from inelastic scattering from sources localized within the crystal (*e.g.* core excitations). It is also noteworthy that the fringe detail arises in the low angle scattering, and the asymmetry arises in the high-angle scattering (compare C and D in Fig. 3). Asymmetry of the 000 beam is high, and at 50kV, exceeds that of the elastic pattern. This shows the influence of the 'accidental' lines. As seen in the underexposed pattern of Fig. 1(*c*) these lines cause increased asymmetry from dynamic interference, and this influence is diffused in the background scattering and so is always involved in the background intensity. This emphasizes the difficulties associated with interpreting absorption asymmetries from bent crystals in the electron microscope, where these lines are not resolved.

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* The terms Kossel and Kikuchi are used in their usual sense: Kossel lines are formed by an external convergent or divergent source; Kikuchi lines are produced by scattering sources within the crystal.

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Formation of rhombohedral polytypes of cadmium iodide.

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Theoretical stacking-fault energies of all known rhombohedral structures of cadmium iodide have been calculated. It has been found that the polytypes occurring more frequently do not possess minimum energies. It can be explained only if mixed dislocations of Burgers vectors ($\mathbf{c}+\mathbf{a}/3+2\mathbf{b}/3$) are considered responsible for the creation of rhombohedral polytypes in cadmium iodide.

Out of nearly 160 polytypes of CdI$_2$ reported so far (Trigunayat & Chadha, 1971), fourteen possess rhombohedral structures. The complete structures of only 7 of them, $12R$, $24R$, $30R$, $36R$, $42R$, $60R$ and $72R$, have been determined. Their formations can be thought of as resulting from the common structure $4H$, due to the occurrence of a mixed dislocation made up of a screw dislocation of Burgers vector $(n/3)\mathbf{c}$ along the c axis and an edge dislocation of Burgers vector ($\mathbf{a}/3+2\mathbf{b}/3$) in the basal plane. The structure of a crystal is affected by the Burgers vector of the screw dislocation alone. When it is an integral multiple of the height of the unit cell of the basic structure $4H$, the resulting structure will evidently belong to a polytypic rhombohedral series $[(22)_n\ 13]_3$. On the other hand, a screw dislocation of Burgers vector $(2n+1)\mathbf{c}/2$, which is a nonintegral multiple of the height of the basic unit cell, will give rise to another polytypic series $[(22)_n\ 1212]_3$. Since the former type of screw dislocations has the layers in perfect fit with each other in contrast to the latter type which produces a misfit, the former should occur more frequently