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ABSOLUTE STRUCTURE DETERMINATION USING ELECTRON MICROSCOPY

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When the symmetry operations, which constitute the space group of a structure, do not include an inversion or a reflection operation, then the structure can exist in two forms, a right-handed and a left-handed one, called enantiomorphs. The presence of the two enantiomorphs coexisting within a sample can be verified in the electron microscope by imaging in dark field in a multi-beam orientation, with the electron beam parallel with a zone axis, along which the crystal does not show a center of symmetry in projection (1). One takes advantage here of violations in Friedel's Law (2), which may cause a difference in background intensity between the two structures. An example is shown in Fig. 1, where an inversion boundary runs through a thick wedge-shaped crystal of lithium ferrite. Ordered lithium ferrite may occur in two enantiomorphic forms $P4_132$ (right-handed screw axis), and $P4_332$ (left-handed screw axis). In this paper it will be shown that it is possible to determine uniquely the configuration of the structure using the results of the dynamical theory.

Thickness fringe profiles were calculated for both structures using the Howie-Whelan equations, generalised for the n-beam case and rewritten for the case of a non-centrosymmetric crystal. The phase factor of the extinction distances and absorption lengths were assumed to be the same (2). The result corresponding to the orientation of the crystal in Fig. 1 is shown in Fig. 2. In the thicker portions of the crystal the fringe pattern is quite sensitive to orientation and the values of the absorption parameters. However, it is noted that the first bright fringe will be closer to the edge of the foil in the $P4_3$ variant than in $P4_1$ for $g = \bar{1}10$. This result proved to be valid for a wide range of deviation parameters and values of the absorption parameter. The latter is understandable because in thin crystals absorption can be neglected. Using this result, it is clear that in Fig. 1 domain 1 has the right-handed $P4_132$ configuration and domain 2 the left-handed $P4_332$. When performing this analysis, care has to be taken to correctly index the diffraction pattern. The calculations were performed with atomic coordinates referred to right-handed axis, standard in crystallography. In the positively printed diffraction pattern however, a left-handed reference system has to be used then for indexing.

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2. R. Serneels, M. Snykers, P. Delavignette, R. Gevers, S. Amelinckx, *phys. stat. sol. (b)*, 58, 277 (1973).

Fig. 1

XBB 764-3193

$1\bar{1}0$ dark field of an ordered LiFe_5O_8 crystal. Voltage 650kV.
In domain 2 the first bright fringe is closest to the edge (60,000x)

P4332 43-BEAM CASE UUV=332 SYMMETRIC KX=0./0./0.

P4132 43-BEAM CASE UUV=332 SYMMETRIC KX=0./0./0.

Fig. 2

XBL 764-1248

Calculated thickness fringe profiles for P4.32 (a) and P4.32 (b) for $g = 110$. The maximum intensity is $.2I_0$. The maximum thickness is $5\xi(440)$. $\xi(440) = 992\text{\AA}$. $\xi'_0 = \xi'_g$; $\xi_g/\xi'_g = .1$ for all g . 43 beam calculation.

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