

Refinement of twinned crystal data

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Abstract. The refinement of structure from diffraction intensities measured from a twinned crystal involves the evaluation of intensity contributions from the various twin domains in the crystal and the corresponding structure factor derivatives. The modifications in the standard least-squares refinement program required for analysis of data from a twinned crystal sample are discussed. The procedure can be extended to cases involving more than two types of twin domains or to samples having mixed phases. The results and limitations of structure refinement using data obtained from a twinned LiKSO_4 crystal in various low-temperature phases are illustrated.

Keywords. Twinned structures; pseudosymmetry; intensity addition; mixed phases.

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1. Introduction

Twinning implies the symmetrical union of two crystallites at a composition plane, the two being related by reflection across a plane or rotation about an axis. According to Mallard's empirical theory (Buerger 1945), twinning is probable in a crystal species if the lattice satisfies certain symmetry requirements. Thus if the cell of the lattice or the supercell has higher symmetry than the point group symmetry of the crystal, twinning is likely. Buerger (1947) noted that twins occur with greater frequency in a derivative structure i.e. one which has been derived from a basic structure by suppressing one or more of its symmetry operations.

In diffraction experiments, especially in the study of structural phase transitions, one is frequently forced to record data from twinned samples arising especially in derivative structures. Further, quite often it is not possible to identify the existence of twins from the diffraction photographs particularly in cases involving 'merohedral' twinning (Buerger 1945) or pseudosymmetry. Twins can normally be detected optically under a polarizing microscope if the crystal is non-cubic. Twins can also be detected in diffraction experiments if they show splitting of diffraction profiles or split spots in x-ray photographs. Thus, if the two lattices are sufficiently misoriented, they can be resolved. If however there is no angular misorientation between the crystallites, i.e. if the reciprocal lattices of the twin domains coincide with each other ($\omega = 0$, twins of merohedry), the twins cannot be detected by any of the aforementioned methods. In such a case, although the magnitudes of the structure factors due to the various crystallites may be different yet the physical detection of twins is elusive. The twins