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Twinned Crystals. IV. $TlNO_2.4tu$* and $Tl_2CO_3.8tu$

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Complicated *hk*0 Weissenberg photographs from orthorhombic $TlNO_2.4tu$ (tu = thiourea) have been explained in terms of twinning on (110) and (1$\bar{1}$0) planes (three individuals). The more complicated photographs from (isomorphous) $Tl_2CO_3.8tu$ have been explained in terms of twinning on (110), (1$\bar{1}$0) and (120) planes (six individuals).

In previous papers in this series twinning has been shown to provide an explanation for the complicated diffraction patterns obtained from 10-methyl-1,2-benzanthracene (Herbstein, 1964) and α-1,2,4,5-tetrachlorobenzene (Herbstein, 1965*a*) and for the deceptively simple diffraction patterns from γ-*o*-nitroaniline (Herbstein, 1965*b*). We present here interpretations in terms of twinning of the similar but not identical diffraction patterns of the isomorphous orthorhombic crystals $TlNO_2.4tu$ and $Tl_2CO_3.8tu$ (Boeyens & Herbstein, 1967). These authors determined cell dimensions and space group for $TlNO_2.4tu$ from oscillation and Weissenberg photographs; the Weissenberg photographs showed fairly complicated domain effects which were noted but not explained. The Weissenberg photographs from $Tl_2CO_3.8tu$ were so complicated that Boeyens & Herbstein (1967) were forced to use powder patterns for determination of cell dimensions and space group.

We now explain the diffraction patterns from these two substances in terms of multiple twinning. The crystals were prepared following Boeyens & Herbstein (1967).

The *hk*0 and *hk*1 Weissenberg photographs from $TlNO_2.4tu$ had a similar appearance. The *hk*0 reflexions

* tu = thiourea

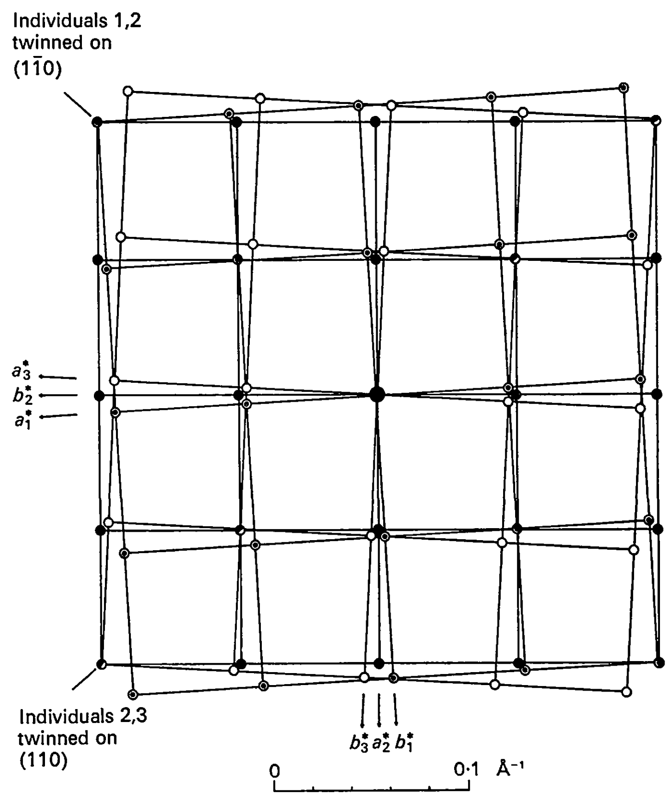

Fig. 1. The relation between the reciprocal lattices of the three individuals found in $TlNO_2.4tu$.

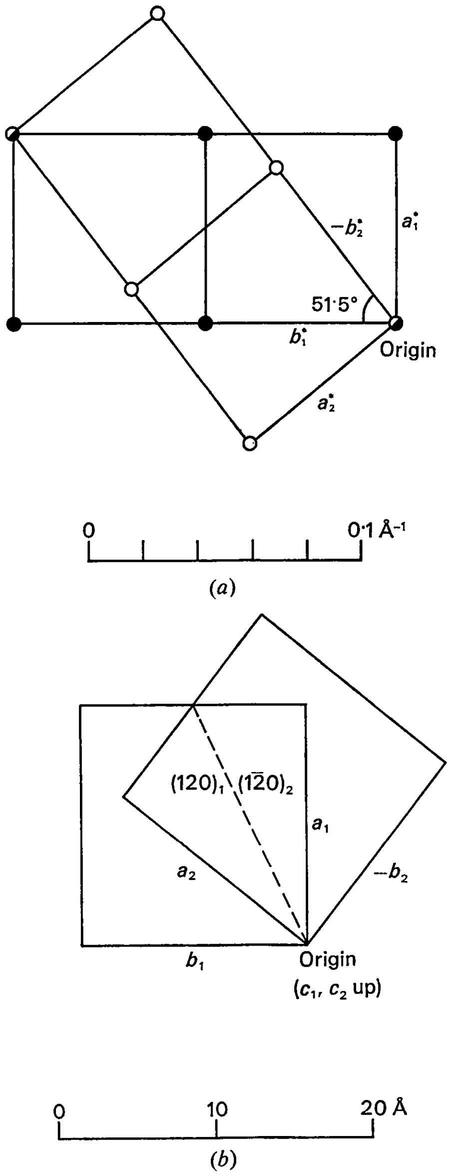

Fig. 2. The relation between the two groups of lattices found in Tl_2CO_3.8tu, (*a*) in reciprocal space, (*b*) in direct space.

belonging to the reciprocal lattice of a single individual were first identified; it was then possible to identify the *hk*0 reciprocal lattices of two other individuals and these three patterns together accounted for all the reflexions. The three reciprocal lattices are shown in Fig. 1. The following cell dimensions and space group, derived from oscillation and Weissenberg photographs, are in good agreement with the earlier results:

a = 14·69, b = 13·93, c = 8·33 Å; space group *Cccm*;
4 formula units per cell.

The three individuals have a common [001] axis and essentially equal volumes; the first and second are twinned about $(1\bar{1}0)$ and the second and third about (110).

The Tl_2CO_3.8tu Weissenberg photographs were similar to those from $TlNO_2$.4tu but more complicated. Identification of a diffraction pattern from a single individual led to the following crystallographic results:

a = 14·83, b = 14·28, c = 8·45 Å; space group *Cccm*;
2 formula units per cell.

The agreement with the values deduced from powder patterns (a = 14·41 (misprinted as 14·14), b = 13·87 Å) (Boeyens & Herbstein, 1967) is rather poor.

Further analysis of the photographs led to identification of twinning about (110) and $(1\bar{1}0)$, as in $TlNO_2$.4tu. However this accounts for only half of the reflexions in the photograph, as there is another group of three individuals, twinned on (110) and $(1\bar{1}0)$ as before but rotated by 52° with respect to the first group about their common (001) axis. This corresponds to additional twinning on (120) (Fig. 2). Thus the Weissenberg photograph has *hk*0 reciprocal layers from six individuals, twinned as described above on (110), $(1\bar{1}0)$ and (120).

The Tl_2CO_3.8tu crystal examined consists of two parts, related by twinning about (120), and of appreciably different volumes (say 3:1). Each part further consists of three individuals, with equal volumes and related by twinning about (110) and $(1\bar{1}0)$. The two complexes twin in similar fashion about {110}; twinning on (120) was not found in $TlNO_2$.4tu.

Macroscopically-twinned crystals of TlH_2PO_4.4tu (isomorphous with $TlNO_2$.4tu and Tl_2CO_3.8tu) have been obtained by Verhoef & Boeyens (1968), who determined their crystal structure. Good single crystals were obtained by mechanically splitting the twinned needles (twin plane not stated) along their long axes. The close structural similarity in **a** and **b** directions provides a direct explanation for the occurrence of twinning on (110) and $(1\bar{1}0)$; fine-scale twinning can be expected on these planes. No structural explanation for twinning on (120) is apparent and here larger-scale twinning, based on dimensional resemblances between the lattices (Fig. 2) is anticipated.

References

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