Acta Cryst. (1972). A 28, 472

Room-temperature effect on dislocations in CdI₂ crystals. By V. K. AGRAWAL, Department of Physics, Hastinapur

College, New Delhi-21, India

(Received 19 May 1972)

X-ray diffraction photographs of CdI_2 crystals, grown from solution at room temperature, frequently exhibit streaking, arcing and polytypism. When the crystals were re-examined after three-four years, the streaking and the arcing were found to have changed. These changes are explained in terms of creation of stacking faults due to the dissociation of existing unit edge dislocations into partials and their movements at room temperatures varying from 5 to 45°C during the year.

Introduction X-ray oscillation photographs of CdI₂ crystals frequently

the polytype 48R, out of four are joined vertically by a continuous streak.

Discussion

exhibit the phenomena of arcing, which consists of an extension of the diffraction spots into small arcs or closed rings on the X-ray Laue photographs, and streaking, in which the reflexions on the *l* rows are connected by streaks. The former has been satisfactorily explained in terms of unit and partial edge dislocations created during crystal growth and forming tilt boundaries, and the latter in terms of stacking faults created randomly during growth (Agrawal & Trigunayat, 1969a, b). Recently Lal & Trigunayat (1970, 1971) have treated CdI₂ crystals at temperatures of 240 to 300°C and have observed characteristic changes in streaking and arcing on oscillation photographs. The changes have been explained in terms of the movement of edge dislocations, either already existing inside the crystal or subsequently generated upon heating. Trigunayat (1971) has also mentioned an increase in streaking in a CdI₂ crystal kept at room temperature for more than a decade. The author has re-examined a few CdI₂ crystals three to four years after their growth. In one crystal a sufficient change in arcing and streaking was observed on Laue and oscillation photographs, and the results are presented in this paper.

Experimental methods and results

The crystals re-examined were grown from a supersaturated solution at room temperature. The details of growth procedure, setting of crystals, selection of range of oscillation and angle of incidence for the Laue photographs, and the X-ray methods employed can be found in a previous publication (Agrawal & Trigunayat, 1969*a*).

Figs. 1 and 2 are the Laue and oscillation photographs of a CdI₂ crystal just after its growth was completed. The Laue photograph exhibits a hexagonal ring for each plane of reflexion. The spots on the rings are elongated towards their centres. The oscillation photograph shows four spots for each reflexion with very faint streaking along the layer lines. The spots on an arc are not joined by any streak. Fig. 3 and 4 are the Laue and oscillation photographs of the same crystal after keeping it in a desiccator for nearly 4 years. The room temperature varied in this period from about 5 to 45 °C. The hexagonal shape of the rings does not change. However, on the right side of the ring another triangular ring is formed, shown by arrows in Fig. 3. Fig. 4 shows two changes (cf. Fig. 2): (i) there is an increase in the streaking along the layer lines; (ii) two spots, belonging to The ring formation on Laue photographs has already been explained in terms of six tilt boundaries making an angle of 60° with each other and consisting of vertically aligned unit edge dislocations lying on the different basal planes (Agrawal & Trigunayat, 1969b). The elongation of spots tcwards the centre is the result of the variation of spacing between two consecutive dislocations. The faint streak between the spots on the ring shows the small region of curvature between two blocks separated by a boundary. The weak streaking joining the spots on the layer lines reveals the small percentage of stacking faults in the crystal. The formation of triangular rings on a side of the ring (Fig. 3) is the result of dissociation of some unit edge dislocations of a boundary into two partials, making an approximate angle of 120°. They have formed their own tilt boundaries providing two extra sides of the triangular ring. These partials cannot move out of the crystal as their separation depends upon the stacking fault energy of the material: the greater the stacking fault energy, the less will be the separation between the partials. The effect of triangular rings on the oscillation photograph (Fig. 4) is to give rise to vertical streaks joining the middle two spots on the arcs, and the intrinsic stacking faults lying between the dissociated partials will produce streaking along the layer lines.

These observations show that the stacking faults are created in the crystal even after its growth is complete, as a result of small variations in room temperature. Thus, it appears that the same phenomenon does occur during the crystal growth from solution at room temperature, which varies by about 20 °C. If these faults are created at regular intervals, a polytype is formed. The mechanism determining the distribution of stacking faults and the influence of the conditions of crystal growth are not yet known.

References

AGRAWAL, V. K. & TRIGUNAYAT, G. C. (1969a). Acta Cryst. A 25, 401.

AGRAWAL, V. K. & TRIGUNAYAT, G. C. (1969b). Acta Cryst. A 25, 407.

LAL, G. & TRIGUNAYAT, G. C. (1970). Acta Cryst. A 26, 430. LAL, G. & TRIGUNAYAT, G. C. (1971). J. Cryst. Growth, 11, 177.

TRIGUNAYAT, G. C. (1971). Phys. Stat. Sol. (a), 4, 281.



Fig. 1. Laue photograph of a CdI₂ crystal. Incident radiation makes an angle of $25^{\circ}40'$ with the *c* axis; *a* axis vertical, Cu white radiation; camera radius 3 cm. Diffraction spots in reflexion alone are shown.



Fig. 2. *a*-axis 15° oscillation photograph of the crystal in Fig. 1. The angle between the incident beam and the *c* axis varies between 25 and 40°. Cu $K\alpha$ radiation; camera radius 3 cm.



Fig. 3. As Fig. 1 but after 4 years.



Fig.4. As Fig.2 but after 4 years.