Acta Cryst. (1962). **15**, 1045

Twist in whiskers revealed by the dynamic fine structure in electron diffractograms.

By Jon Gjønnes, *Division of Chemical Physics, C.S.I.R.O. Chemical Recearch Laboratories, Melbourne, Australia**

(*Received* 25 *February* 1962)

During a study of the dynamic fine structure from needle-shaped crystals of sodium chloride some observations were made which indicated large twists in some of the crystals. The purpose of this note is to show how a twist may reveal itself in the recorded fine structure. The needle-shaped crystals, which were grown in solution, or by evaporation of NaCl-solution on microscope grids in a moist atmosphere, were frequently bent, and gave readily several strong diffraction spots, although the crystals were relatively thick, usually $0{\cdot}5\mu$ or more, indicating some spread in orientation within the needle. Fig. 1 shows part of a diffraction pattern—the layer line (hhh) from a whisker $0{\cdot}5\mu$ thick whose axis was ca. 72° from the electron beam.

The most conspicuous features of the pattern are that (i) the dynamic splitting of the reflexions shows only two spots, instead of the four expected from a thick crystal, (ii) the direction of separation of the spots varies systematically with the reflexion, (iii) the layer line is slightly curved.

If the doubling of the spots is caused by refraction, a simple geometrical model can be given which explains the observations. A whisker containing one or more screw dislocations parallel to its axis will have different orientation for diametrically opposite sides, the orientation difference being approximately $\alpha = b/d$, where b is the Buergers vector and d the diameter. Further, the orientation changes with position along the whisker axis, due to the axial twist $\beta \sim b/d^2$, so that the reciprocal-lattice points corresponding to the two sides will be spread out on two arcs slightly shifted from each other. For a straight whisker, these two arcs, l and l', will lie in a plane normal to the whisker axis. When the whisker is also bent, as was seen from low-magnification micrographs taken in the diffraction camera, l and l' will be approximately normal to an axis **e** describing the combined effect of bend and axial twist (see Fig. 2). The directions of the effective reciprocal-lattice vectors for the two sides will be determined by the intersections, A, A', of these arcs with the Ewald sphere and will generally correspond to different positions along the whiskers. The curvature of the Ewald sphere causes the intersections to be at different heights above the plane normal to the incident beam, and the layer lines will be

* Present address: University of Oslo, Department of Physics, Blindern, Oslo, Norway.

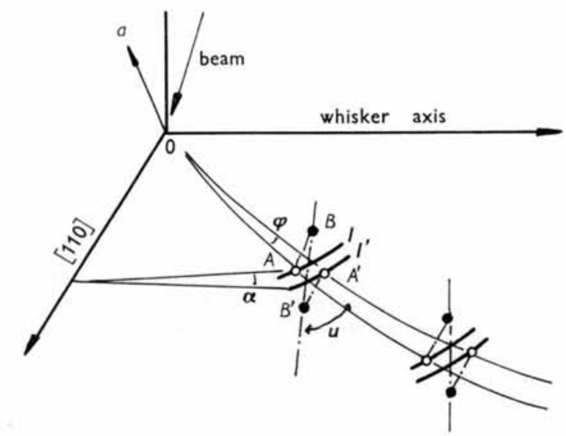

Fig. 2. The arcs l and l' are loci for the end points of the reciprocal lattice vector (111) for the two sides of the whisker; l and l' lie in two planes through 0, approximately normal to the axis **e** describing the change in orientation along the whisker, i.e. the combined effect of bend and axial twist. A, B, etc. are points on the Ewald sphere; A, A' are the intersections of the Ewald sphere and l, l', and determine the directions of the (111) diffracted beams from the two whisker sides, neglecting refraction. $AB = A'B' = \boldsymbol{\delta}/2$ is the refraction displacement; $\boldsymbol{\delta}$ is normal to the whisker axis. B, B' determine then the observed, refraction displaced, spots. $\varphi = \angle AOA'$, $u_0 = \angle OAB$, $u = \angle (OA), (BB')$. Note that the distance AA' increases with increasing distance from the origin, whereas the refraction displacement $\boldsymbol{\delta}/2$ is constant; this is the essential reason for the variations in the direction of the observed spot separation, BB'.

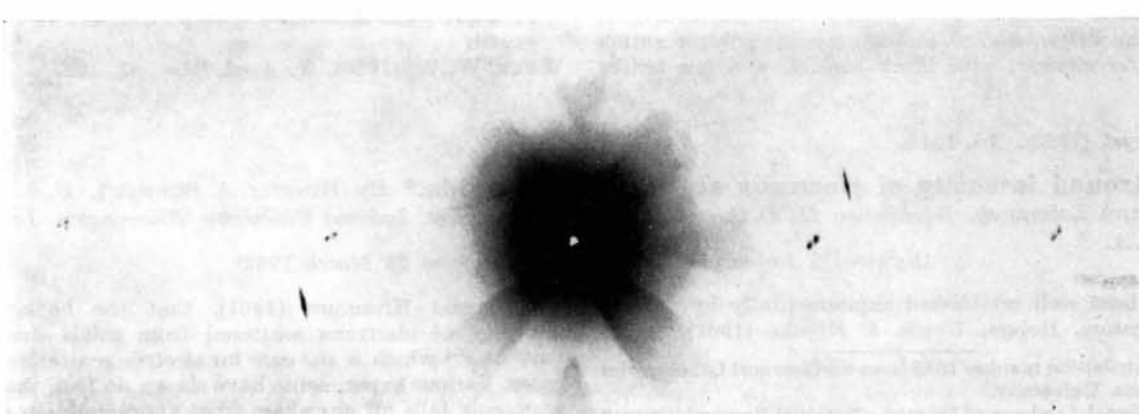

Fig. 1. Electron diffraction pattern from NaCl whisker: (hhh) line of spots, beam near $[4\bar{2}\bar{2}]$-direction.

Table 1. *Observed and corrected refraction displacements*

Reflexions	222	111	$\bar{1}\bar{1}\bar{1}$	$\bar{2}\bar{2}\bar{2}$	$1\bar{1}3$	$\bar{1}1\bar{3}$	204	$\bar{2}0\bar{4}$
Δ (mm.)	1·5	1·5	1·5	1·7	2·75	1·8	2·5	1·8
u (°)	25	30	35	40	50	−15	50	−30
$\Delta \cos u$ (mm.)	1·4	1·3	1·2	1·4	1·8	1·7	1·6	1·6
u_0 (°)	37				20		10	
$\delta=\Delta \cos u/\cos u_0$ (mm.)	1·55				1·85		1·60	
φ (radians)	8.10^{-3}				10^{-2}		8.10^{-3}	

curved, when the angle between **e** and the whisker axis is different from 90°.

As illustrated in Fig. 2, refraction will further shift the two spots in opposite directions, normal to the projection of the whisker axis. If the twist and bending are homogeneous, the refraction component, δ, of the separation can be calculated from the magnitude, Δ, and the direction, u, of the observed separations. Results are given in Table 1. u_0, the angles between the reciprocal-lattice vectors and the refraction displacement, correspond to the assumed average orientation, $[4\bar{2}\bar{2}]$.

The resulting δ is appreciable higher than the value 1·2 calculated from the Fourier coefficient V_0 and the orientation $[4\bar{2}\bar{2}]$ assuming two-beam conditions.

From the measured curvature of the layer line and the bending ($\sim 2.10^{-2}/\mu$) observed in the micrograph, an estimated lower limit of the axial twist, $\sim 10^{-2}/\mu$, was obtained, corresponding to a Buergers vector of $\gtrsim 20$ Å. The angular difference between the reflecting positions of the two sides can then be translated into an upper limit of the side-twist. We obtain $\alpha \sim 5.10^{-3}$ corresponding to $b \lesssim 25$ Å for a central dislocation.

As mentioned above, the diffracting parts of the two sides of a whisker, e.g. the parts corresponding to (111) having the orientations OA and OA' in Fig. 2, will have different positions along the whisker axis. In a low-magnification dark-field image from another, straight, whisker, such a position difference between the diffracting parts was actually observed. Assuming the axial twist to be b/d^2 and the side-twist to be b/d, the expected shift in position is readily found to be h_2/h_1 diameters, where h_1 and h_2 are the components of the diffraction vector along the normal to the whisker axis. The observed shift was nearly twice as large, indicating either a side-twist larger than b/d, as may be the case when the screw dislocation is non-central, or an axial twist lower than the theoretical value.

Some of the whiskers appeared to have even larger twist than the one described here, and streaking of the spots, as is visible in some of the reflexions in Fig. 1, was frequently observed, indicating some polygonization along the whisker, with block lengths of a few tenths of a micron and orientation differences of the order of 5.10^{-3}.

The lack of the usual dynamic splitting in two separate waves from each of the wedges is not fully understood. The implication is, of course, that the equal-thickness fringes from the crystal wedges have only slight contrast or irregularities in spacing so great that their Fourier representation by two components breaks down. The latter case may be realized through many-beam (discrete *or* continuous) interactions, but it appears more likely that the effect is directly connected with the mentioned lattice twist, which will cause the excitation error, ζ, for planes at an angle with the whisker axis, to vary with distance from the edge. By considering the argument $(\frac{1}{2}H(\zeta^2+V_h^2/k^2)^{\frac{1}{2}})$ of the pendulum solution (see e.g. Pinsker, 1953; H, ζ, V_h and k have their usual meaning) one finds that the lateral periodicity of fringe contrast may be appreciably disturbed for twists of the order $10^{-2}/\mu$ or more. Similar qualitative conclusions may be reached by studying the projected potential of a twisted crystal and applying the 'phase-grating approximation', which is known to predict the correct dymanic fine structure (Cowley & Moodie, 1961).

It should be noted that large dislocation densities in sodium–chloride whishers have been reported previously (Webb, 1960).

My thanks are due to Dr J. M. Cowley for suggesting this problem and for valuable advice. Financial support from the Commonwealth Scientific and Industrial Research Organization and the Royal Norwegian Council for Scientific and Industrial Research is gratefully acknowledged.

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Acta Cryst. (1962). **15**, 1046

Background intensity of electrons scattered from solids.*

By Russell A. Bonham†, *U.S. Naval Research Laboratory, Washington 25, D.C. and Chemistry Department, Indiana University, Bloomington, Indiana, U.S.A.*

(*Received* 2 *January* 1962 *and in revised form* 26 *March* 1962)

It has been well established experimentally by various investigators, Hongo, Uyeda & Miyake (1961), Bauer (1962), and Kitamura (1961), that the background intensity of electrons scattered from solids does not vary as s^{-4} which is the case for electron scattering from gases. Various experiments have shown, in fact, that this scattering falls off anywhere from approximately s^{-2} all the way up to s^{-1} or higher. It is the purpose of this note to point out that in the limit of thin film thickness,

* Contribution number 1045 from theChemical Laboratories of Indiana University.

† National Academy of Science—National Research Council—U.S. Naval Research Laboratory Research Associate for the year 1960.