1000 reflexions contains only about 150 general reflexions with $S/\Sigma = 1$ and the unweighted intensities would have a specific variance (Wilson, 1951) of intrinsic value 3·6. The salt $NaK_5Cl_2(S_2O_6)_2$ had alternative space groups $P4nc$ or $P4/mnc$ (Stanley, 1953). The $N(z)$ distributions (Howells *et al.*, 1950) for the [100] zone with and without proper weighting of the intensities are shown in Fig. 1.

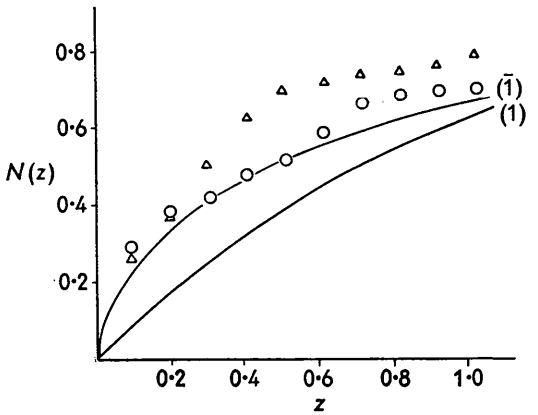

Fig. 1. $N(z)$ distribution for reflexions in the [100] zone of $NaK_5Cl_2(S_2O_6)_2$. Triangles: unweighted intensity data; circles: properly weighted intensity data.

The values of the specific variance with and without proper weighting were 2·14 and 2·34.

Since the distribution of intensities in a group of reflexions heterogeneous in S/Σ is a function of $|\mathbf{s}|$, failure properly to weight the intensities may result in wrong conclusions concerning pseudo-symmetry.

In the method of multipliers (Rogers, 1950) the values of S and Σ are compared and it is necessary that the correct weighting procedure be observed in the determination of the value of Σ(obs.).

Conclusion

The distribution functions of Wilson (1949) apply to the intensities in any group of reflexions for which the value of S/Σ is the same for all reflexions and which is homogeneous in distribution type. In the determination of the temperature coefficient and the factor for conversion to the absolute scale all reflexions should be weighted by Σ/S or omitted if this quantity is not known. Groups of intensities used for the statistical tests for symmetry elements must be homogeneous in distribution type and in S/Σ or made so by weighting by Σ/S.

References

HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* **3**, 210.
KARTHA, G. (1953). *Acta Cryst.* **6**, 817.
LIPSON, H. & WOOLFSON, M. M. (1952). *Acta Cryst.* **5**, 680.
ROGERS, D. (1950). *Acta Cryst.* **3**, 455.
ROGERS, D., STANLEY, E. & WILSON, A. J. C. To be published.
ROGERS, D. & WILSON, A. J. C. (1953). *Acta Cryst.* **6**, 439.
STANLEY, E. (1953). *Acta Cryst.* **6**, 187.
WILSON, A. J. C. (1942). *Nature, Lond.* **150**, 152.
WILSON, A. J. C. (1949). *Acta Cryst.* **2**, 318.
WILSON, A. J. C. (1951). *Research, Lond.* **4**, 141.

Acta Cryst. (1955). **8**, 357

The application of an X-ray method to the study of lattice bending.

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The limit of resolution of many of the X-ray techniques employed in the study of the mechanism of sub-boundary formation is such that much of the evidence obtained, such as the background darkening between separated X-ray diffraction spots, may be interpreted as arising either from a continuously bent lattice or from sub-grains that are too small to be resolved and whose orientations cover the range between the resolved X-ray spots (Beck, 1954). The high resolving power of the double-crystal diffractometer, together with the Berg–Barrett technique of X-ray microscopy, gives detailed information which enables one to eliminate the possibility of lattice bending.

The rocking curve obtained using Cu $K\alpha$ radiation reflected in the first order from the cleavage planes of calcite mates mounted as the A and B crystals of the diffractometer had a half-width of 14 seconds of arc. The half-width of the first crystal is therefore $14/\sqrt{2} \approx 10$ seconds of arc. Crystal B was then rotated to the $(1, +1)$ position and the slit system was adjusted until most of the α_2 component of the rocking curve was suppressed (Intrater & Weissmann, 1954). The axis of rotation coincides with what in the experiment is the specimen surface, and the angular rotation is so small that the area of the specimen irradiated can be considered to be fixed.

A rocking curve of the test specimen mounted in the $(n, -n)$ position is obtained and is then retraced in discrete steps with an image of the reflecting area recorded at each setting. The small area of specimen irradiated will register, on a film placed close to and parallel to the reflecting planes, an image that consists of reflection from all regions accessible to the beam whose orientations are identical within the 10 seconds of arc non-parallelism of the monochromatized beam; misoriented neighbors will reflect to adjacent regions on the film but will do so at different specimen settings (Intrater & Weissmann, 1954).

Consider a specimen containing a random distribution of relatively perfect unresolvable small domains whose overall misorientation is 40 seconds of arc. The peak of the Gaussian rocking curve and the densest image are recorded for that specimen orientation in which the

greatest number of domains are simultaneously reflecting. The image recorded at each stationary setting would be of uniform intensity and would equal the area irradiated since there are, throughout that area, domains that simultaneously fulfill the Bragg condition. The bent lattices of Fig. 1 and the sequence in which their images

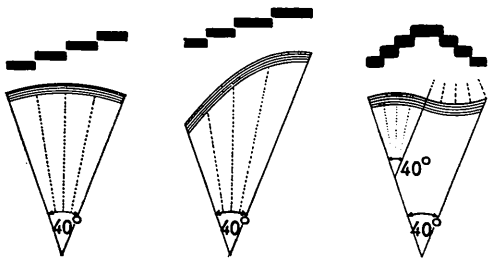

Fig. 1. Schematic representation of the correlation between lattice bending and X-ray images.

are recorded illustrate that, if the lattice is bent, it will not be possible to record the complete irradiated area in one setting. The minimum number of specimen settings required will equal the angular range of reflection divided by the horizontal divergence of the beam. Four settings would be required for each of the examples of Fig. 1 since the reflection range is 40 seconds of arc and the beam imperfection is 10 seconds of arc.

The ability of the technique to rule out the possibility of lattice bending and to distinguish between regions of slightly different orientation was established by data obtained from a fixed area of a zinc single crystal grown from the melt and subsequently quenched and annealed. The (0001) reflection from a face acid-cut parallel to the cleavage planes was examined. The correction due to dispersion was negligible in the $(n, -n)$ position. As grown, the rocking curve was Gaussian with a half-width of 40 seconds of arc and the image recorded with the specimen stationary at the rocking curve peak was uniform in intensity and identical in area and shape to the area irradiated and to the image recorded by continuously rotating the specimen throughout its angular range of reflection. Images recorded at the specimen orientations of half maximum intensity were of the same area as that recorded at the peak. The specimen condition is therefore similar to the random condition described, and there is no evidence of lattice bending.

The specimen showed evidence of damage when quenched from 400° C., for the rocking curve half-width was then 6 minutes of arc and the reflecting range 14 minutes of arc; such an increase was not observed for other zinc crystals when quenched. Annealing at 400° C. for 1 hr. broke this rocking curve up into one having several peaks and a total reflection range of 8 minutes of arc. Only seven stationary exposures were required to record images whose sums equal the area irradiated and that recorded by continuous rotation. More than seven sub-grains were observed since several of the images recorded simultaneous reflection from non-adjacent domains having the same orientation. The possibility of lattice bending was, therefore, eliminated and the possibility that the lattices of the individual sub-grains were bent was eliminated by the observation that there was, for each sub-grain, an orientation that gave an image of maximum area and that this area encompassed the images recorded for that particular sub-grain in slightly different angular positions.

The high resolving power of the technique gives detailed information about the lattice topography. Within the limitations imposed by the imperfection of the first crystal, the possibility that specimen lattices are bent can be eliminated by the observation that the image size and shape of one setting, or a number of settings less than that calculated by dividing the reflecting range by the imperfection of the monochromatized beam, is equal to the area irradiated.

References

BECK, P. A. (1954). *Advances in Phys.* **3**, 245.
INTRATER, J. & WEISSMANN, S. (1954). *Acta Cryst.* **7**, 729.

Acta Cryst. (1955). **8**, 358

The conformation of 1:4-diphenyl-1:4-diarsa*cyclo*hexane.

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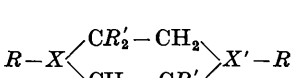
F. G. Mann and his co-workers (Beeby & Mann, 1951; Mann & Millar, 1952; Jones & Mann, 1955) have prepared and examined a number of heterocyclic compounds



(I)

of type (I), in particular those in which $X, X' =$ N, P or As (including those where $X \neq X'$). These compounds show interesting variations in their behaviour towards quaternizing agents, in that certain of them (e.g. (II) and (III)) will readily undergo diquaternization with simple alkyl halides but none will undergo diquarternization (with cyclization) with alkylene dibromides.

Thus 1:4-dimethylpiperazine ($X = X' =$ N, $R = CH_3$, $R' =$ H) reacts with ethylene dibromide to form a diquaternary salt bridged across the nitrogen atoms, but none of the following compounds will quaternize in this manner: