

Molecules are bound together in molecular ribbons along b by N-H \cdots O hydrogen bonds shown schematically in Fig. 4. It is this molecular arrangement that

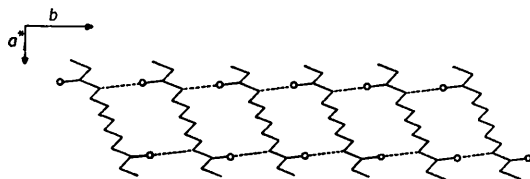


Fig. 4. Schematic representation of molecular ribbon.

results in cutting anisotropy of the crystals, i.e. easy cutting parallel to b but only with difficulty normal to b .

We wish to express our thanks to Mr Charles

Chaney, who assisted in the calculations. We wish to acknowledge financial support from the National Institute of Health contract A-858, the Fund for Biological and Medical Research of the University of Washington and the U.S. Army Quartermaster Corps Contract No. Da 19-129-QM-523.

References

- BAILEY, M. (1955). *Acta Cryst.* **8**, 575.
 CRUICKSHANK, D. W. J. (1949). *Acta Cryst.* **2**, 65.
 JENSEN, L. H. (1956). *J. Amer. Chem. Soc.* **78**, 3993.
 MCGUIRE, T. R. (1949). Ph.D. Thesis, Yale University.
 MCWEENEY, R. (1951). *Acta Cryst.* **4**, 513.
 MADDIN, R. & ASHER, W. R. (1950). *Rev. Sci. Instrum.* **21**, 881.
 WIEBENGA, E. H. & SMITS, D. W. (1950). *Acta Cryst.* **3**, 265.

Short Communications

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Acta Cryst. (1957). **10**, 533

A method of eliminating the primary extinction effect in electron diffraction. By GORO HONJO and NORIHISA KITAMURA, *Tokyo Institute of Technology, Oh-okayama, Meguroku, Tokyo, Japan*

(Received 22 May 1957)

It has been reported by a number of workers that the intensity of electron diffraction by a crystal sometimes deviates from the value calculated by the kinematical theory of diffraction (Yearian & Howe, 1935; Ornstein, Brinkman, Hauer & Tol, 1938; Tol & Ornstein, 1940; Lennander, 1954). Blackman (1939) ascribed it to the dynamical diffraction effect, or the primary extinction effect. On the basis of the experiment of Jamzin (1949), Pinsker (1949) published an opinion contradicting Blackman. Kuwabara (1957), however, recently performed an experiment similar to that of Jamzin on evaporated films of some metals and salts, and recognized that an appreciable extinction effect occurs even for quite small crystallites. Therefore it is very important in electron-diffraction structure analysis to have a method of eliminating the primary extinction effect. One promising method has been recently proposed by Nagakura (1957), utilizing Wilson's intensity statistics combined with the current theory of the primary extinction effect (Blackman, 1939).

Bacon & Pease (1953) gave a method of eliminating the secondary extinction effect in neutron diffraction by the extrapolation of intensities measured at various wavelengths to the wavelength zero. A similar method may be applied also to electron diffraction.

According to the result of the dynamical theory ap-

plied to a parallel-plate crystal (Blackman, 1939), the intensity of a reflexion is given by

$$I \propto f(A) \cdot |F|^2, \quad (1)$$

where F is the structure factor and $f(A)$ is the primary extinction coefficient defined by

$$f(A) = \frac{1}{A} \int_0^A J_0(2x) dx.$$

Here J_0 is the zero-order Bessel function, $A = c|F|H/\lambda$, c is a constant, H is the thickness of the crystal and λ is the wavelength. The function $f(A)$ can be approximated by $\exp(-\frac{1}{3}A^2)$ for $A \leq 2$ within an error less than 1%. Then (1) can be rewritten as

$$\ln I = \ln |F|^2 - \frac{1}{3}c^2|F|^2H^2\lambda^2 + \text{const.} \quad (2)$$

Thus, $\ln I$ should be linear to λ^2 with a gradient $\frac{1}{3}c^2|F|^2H^2$, and the value of $\ln I$ extrapolated to $\lambda = 0$ should give the kinematical value of I .

The above theory may not hold exactly for crystallites of complicated shapes in practical specimens. We can, however, anticipate that the use of the $\ln I - \lambda^2$ plot will give a practical method of obtaining the kinematical intensity.

An experimental observation was carried out for poly-

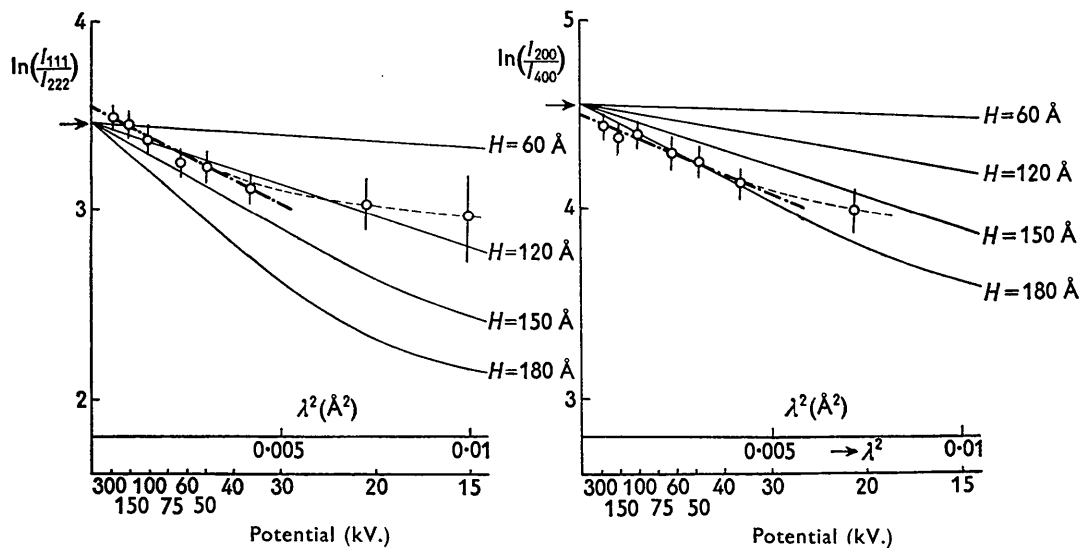


Fig. 1.

crystalline films of aluminium. Films of thickness d of about 200 Å were prepared by evaporation *in vacuo*. The mean crystal size ρ of crystallites in the films was estimated to be 200 ± 30 Å from the width of Debye rings. The accelerating voltage of the electrons was varied from 10 kV. to 240 kV.*, corresponding to the range of wavelength from 0.1 Å to 0.025 Å.

In Fig. 1 the logarithm of the observed intensity ratios, $\ln I_{111}/I_{222}$ and $\ln I_{200}/I_{400}$, are plotted. The pairs of the reflexions of different orders from the same lattice planes were chosen in order to avoid the effect of texture in the films. The vertical line segments represent the probable experimental errors. The theoretical values of the logarithms of the intensity ratios were calculated by (1), assuming a Debye temperature 390° K. (Lonsdale, 1948) and are shown in the figure by full lines for several values of H .

The observed values for shorter wavelengths lie upon the straight chain lines and the extrapolated values coincide with the kinematical values, which are indicated by the arrows, within the experimental errors. The gradients of the two chain lines almost coincide with those of the theoretical lines for the thickness $H = 150$ Å, which is slightly smaller than the observed value of ρ .

The result shows that the linearity of the $\ln I - \lambda^2$ plot does hold, at least approximately, at shorter wavelengths. There is no serious importance in the difference between the values of ρ and H , since their definitions are not identical. It is noted, however, that the observed data for longer wavelengths deviate from the extension of the

chain straight lines more rapidly than it is expected theoretically. A tentative explanation of this discrepancy may be that the effective mean thickness of crystallites decreased rapidly at wavelengths where the absorption became predominant. Since the mean crystal size ρ was almost equal to the specimen thickness d , the secondary extinction effect may be put out of consideration. Further experiments for specimens composed of heavier atoms are now in progress.

In conclusion, the authors express their thanks to Prof. S. Miyake and Mr S. Nagakura for their interest and discussions. The authors' thanks are also due to Dr B. Tadano for providing facilities in the high-tension experiments and to Messrs S. Katagiri and K. Ichige for their assistance in the experiments.

References

- BACON, G. E. & PEASE, R. S. (1953). *Proc. Roy. Soc. A*, **220**, 397.
 BLACKMAN, M. (1939). *Proc. Roy. Soc. A*, **173**, 68.
 JAMZIN, I. I. (1949). *Trudy Inst. Krist. Akad. Nauk SSSR*, **5**, 69.
 KUWABARA, S. (1957). *J. Phys. Soc. Jap.* In preparation.
 LENNANDER, S. (1954). *Ark. Fys.* **8**, No. 54, 551.
 LONSDALE, K. (1948). *Acta Cryst.* **1**, 142.
 NAGAKURA, S. (1957). Unpublished.
 ORNSTEIN, L. S., BRINKMAN, H., HAUER, A. & TOL, T. (1938). *Physica*, **5**, 693.
 PINSKER, Z. G. (1949). *Diffraktsija elektronov*. Moscow: Academy of Sciences. (*Electron Diffraction*. London: Butterworths. 1953).
 TADANO, B., SAKAKI, Y., MARUSE, S. & MORITO, N. (1956). *J. Electronmicroscopy, Jap.* **4**, 5.
 TOL, T. & ORNSTEIN, L. S. (1940). *Physica*, **7**, 685.
 YEARIAN, H. J. & HOWE, J. D. (1935). *Phys. Rev.* **48**, 381.

* For the experiments above 100 kV. a high-tension electron-microscope diffraction unit in the Hitachi Central Laboratories (Tadano, Sakaki, Maruse & Morito, 1956) was used.

Yearian & Howe (1935) observed that the intensities of Debye rings from gold and silver tended to the kinematical values with increasing accelerating voltages (10 kV.-80 kV.).