

**Moiré pattern produced by electron bombardment of a molybdenum trioxide single crystal.** By P. K. GARG, JITENDRA KUMAR and D. L. BHATTACHARYA, *Department of Physics, Banaras Hindu University, Varanasi 5, India*

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A single crystal of molybdenum trioxide bombarded by an intense beam of electrons in an electron microscope developed a new internal structure showing localized fringe patterns in different parts. The phenomenon is explained as being the result of recrystallization of MoO<sub>3</sub> up to only a certain depth in the crystal. The fringe pattern is a rotation moiré fringe pattern resulting from a slight misorientation. This interpretation is confirmed by selected area diffraction patterns.

During an electron microscopic study of molybdenum trioxide crystals obtained from the vapour produced on heating molybdenum metal in air, an interesting phenomenon has been observed.

A single crystal of MoO<sub>3</sub> was selected and its diffraction pattern was recorded; only the second condenser lens was on in this case and it was considerably overfocused. In order to record the transmission micrograph of the crystal at high magnification the first condenser lens, which was pre-set at a current useful for high resolution electron microscopy, was also switched on. When the first condenser was switched on the beam intensity at the specimen suddenly passed through a maximum and the image became very bright. The crystal of MoO<sub>3</sub> (which was previously almost uniformly transparent and a single crystal) developed a new internal structure, while the outline of the crystal remained exactly the same. This irradiated crystal gave a diffraction pattern composed of diffraction spots each of which was split into several closely spaced spots (Fig. 1). This indicated that the surface of the crystal had recrystallized and that the crystallites so formed were misoriented by only small angles.

The corresponding electron micrograph shows a moiré fringe pattern (Fig. 2). The fringes can be seen to be localized at different parts of the crystal and have variable fringe widths and orientations. Since a moiré pattern can only be obtained from two overlapping crystals, a possible explanation for the present case is that there was recrystallization only up to a certain depth in the crystal. The moiré pattern is then the result of the slight misorientation between the parent crystal and the newly formed crystallites.

It has been reported by Hashimoto (1954) that electron irradiation can change the chemical composition of a crystal. A new oxide of molybdenum might have been formed up to a certain depth of the parent crystal. This possibility is ruled out, however, by the electron diffraction pattern of the irradiated crystal. None of the oxides of molybdenum have a lattice vector which is close in magnitude to that of MoO<sub>3</sub> (Pearson, 1967). The presence of these oxides

would produce diffraction spots at significantly different radial distances from those of the parent oxide. In the present case the irradiation-produced spots are found to be simply displaced by small angles, which corresponds to there having been no change in the lattice vector. Moreover, of the secondary diffraction spots, at least two (marked with arrow) can be seen clearly around the direct-beam spot, which is characteristic of a rotation moiré pattern (Dowell, Farrant & Rees, 1956).

The minimum fringe-width observable is about 20 Å and the maximum angle of rotation between two spots in the diffraction pattern is about 10°. The fringe width of a moiré pattern produced by two overlapping MoO<sub>3</sub> crystals in the (010) orientation and with an angle of misorientation of 10° can be calculated to be of the order of 20 Å. This confirms that the moiré fringes are a result of overlapping MoO<sub>3</sub> crystals and not attributable to other causes. Since the external features of the crystal are unchanged, this again confirms that recrystallization has taken place only up to a certain depth of the parent crystal. It is known that MoO<sub>3</sub> vapour-condensed crystals are not always perfect and often have a lineage structure (Heidenreich & Sturkey, 1945). It is possible that these crystals are in a state of strain as a result of warping or other causes and that the stresses are locally partially relieved by thermal activation from the electron beam. The recrystallized regions apparently show epitaxy with the crystal lattice of the parent crystal.

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**Statistical variance of line-profile parameters: Addendum.** By A. J. C. WILSON, *Physics Department, The University of Birmingham, P.O. Box 363, Birmingham 15, England*

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The covariance of a Fourier sine coefficient with a Fourier cosine coefficient takes three different forms, depending on whether the order of the sine coefficient is less than, equal to, or greater than that of the cosine coefficient. Explicit formulae are given.

Roof (1969) has pointed out that the expression given by Wilson (1967, equation 117) for  $\text{cov}_{rs}(A_m, B_n)$ , the covari-

ance of the Fourier sine coefficient of order  $n$  with the Fourier cosine coefficient of order  $m$ , is not valid for  $n=m$ .

Recalculation has shown that the equation as printed contains two errors, and that in its derivation the tacit assumption was made that  $m > n$ . The three cases ( $n < , > ,$  or  $= m$ ) are each readily found from Wilson's equation (116) by substituting  $\sigma^2(I_j) = RI_j/T$  and summing over  $j$ . With the approximation, valid for  $x \ll R$ ,

$$\begin{aligned} \sum j \sin(2\pi xj/R) &= \int_{-\frac{1}{2}R}^{\frac{1}{2}R} j \sin(2\pi xj/R) dj \\ &= 0, \quad x=0, \\ &= (-)^{x+1} R^2 / 2\pi x, \end{aligned}$$

$x$  a positive integer, the covariance for fixed-time counting takes the form:

$$\begin{aligned} \text{cov}_{tt}(A_m, B_n) &= \frac{R}{L_0 T} \left\{ \frac{1}{2} B_{m+n} - \frac{1}{2} B_{m-n} - A_m B_n \right. \\ &+ \frac{(G_R - G_L)R}{2\pi L_0} \cdot \frac{(-)^{m+n} n}{m^2 - n^2} \\ &+ \left. \frac{(G_R - G_L)R}{2\pi L_0} \cdot \frac{(-)^n A_m}{n} + \frac{R_g A_m B_n}{L_0} \right\} \quad (117a) \end{aligned}$$

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**Inter-laboratory single crystal intensity projects.** By S. C. ABRAHAMS,\* L. E. ALEXANDER,\* T. C. FURNAS,\* W. C. HAMILTON,\* J. LADELL,\* Y. OKAYA,\* R. A. YOUNG\* and A. ZALKIN\*†

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Prospective authors of reanalyses of inter-laboratory single crystal intensity projects are urged to contact members of these projects for access to the available unpublished information generated by the projects.

Measurement of identical physical quantities by several laboratories in a collaborative experiment is generally undertaken with a primary emphasis either on achieving higher accuracy in the value of these quantities or else on affording an assessment of the methods themselves. *The American Crystallographic Association Single Crystal Intensity Project* (1967) was developed in terms of the second objective, and basically sought to answer the question: 'How reproducible are structure factors measured diffractometrically in 1964–1965?'. The result obtained was that most of the seven experimental sets of structure factors contained  $|F|^2$  values within five per cent of the mean values and that none agreed much better than to within two per cent; hence, it seems likely that no experiment is closer to the true values than two per cent.

The volume of information generated by this Project was so large that considerable selection was necessary in pre-

\* Bell Telephone Laboratories, Carnegie-Mellon University, Picker Instruments, Brookhaven National Laboratory, Philips Laboratories Inc., State University of New York at Stony Brook, Georgia Institute of Technology, Lawrence Radiation Laboratory respectively.

† The authors are members of the American Crystallographic Association Single Crystal Intensity Project.

for the case  $m > n$ ,

$$\begin{aligned} \text{cov}_{tt}(A_m, B_n) &= \frac{R}{L_0 T} \left\{ \frac{1}{2} B_{m+n} + \frac{1}{2} B_{n-m} - A_m B_n \right. \\ &+ \frac{(G_R - G_L)R}{2\pi L_0} \cdot \frac{(-)^{m+n+1} n}{n^2 - m^2} \\ &+ \left. \frac{(G_R - G_L)R}{2\pi L_0} \cdot \frac{(-)^n A_m}{n} + \frac{R_g A_m B_n}{L_0} \right\} \quad (117b) \end{aligned}$$

for the case  $n > m$ , and

$$\begin{aligned} \text{cov}_{tt}(A_m, B_m) &= \frac{R}{L_0 T} \left\{ \frac{1}{2} B_{2m} - A_m B_m - \frac{(G_R - G_L)R}{8\pi L_0} \cdot \frac{1}{m} \right. \\ &+ \left. \frac{(G_R - G_L)R}{2\pi L_0} \cdot \frac{(-)^m A_m}{m} + \frac{R_g A_m B_m}{L_0} \right\} \quad (117c) \end{aligned}$$

for  $m = n$ . Equation (128) for the covariance arising from the variance of the background has the same form, whatever the relative magnitudes of  $m$  and  $n$  (but is, of course, zero if either  $m$  or  $n$  is zero).

#### References

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paring the report for publication. However, the information not published was always, and is still, regarded as available on request. Similarly, unpublished information generated by the I.U.Cr. Commission on Crystallographic Apparatus Single Crystal Intensity Project (Abrahams, Hamilton & Mathieson, 1969) will also be available on request.

The value to be obtained from reanalyses of such projects would be greatest if made with a full knowledge of the pertinent information. In the recent reanalysis of the *American Crystallographic Association Single Crystal Intensity Project* by Mackenzie & Maslen (1968), a request for such information was not made. It is not our purpose to criticize Mackenzie & Maslen's methods, beyond the comment that elimination of 45 per cent of the data in order to form a 'concordant subset' violates the objective of the A.C.A. project.

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