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An Accurate Absolute Scattering Factor for Silicon

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The 220 Bragg reflexion of silicon has been studied in considerable detail. By the Pendellösung fringe method we have measured the atomic scattering factor with an internal consistency of better than 0·1%. Particular care was taken to exclude systematic errors which might arise from elastic strain, X-ray absorption and X-ray polarization effects. The crystal was cut parallel to the Bragg planes at the points of observation so that its thickness could be directly measured with a travelling microscope. At the value of $\sin\theta/\lambda$ corresponding to the 220 Bragg reflexion, the experimental atomic scattering factors (at 20°C) were: $f=8{\cdot}478\pm0{\cdot}008$ for Mo $K\alpha_1$ radiation; $f=8{\cdot}448\pm0{\cdot}012$ for Ag $K\alpha_1$ radiation, and $f(\text{Mo } K\alpha_1)/f(\text{Ag } K\alpha_1)=1{\cdot}0035\pm0{\cdot}0007$.

Introduction

Several authors have recently made measurements of X-ray structure factors by the Pendellösung method (Kato & Lang, 1959; Hattori, Kuriyama, Katagawa & Kato, 1965; Hart, 1966; Hattori & Kato, 1966; Kato & Tanemura, 1967; Yamamoto & Kato, 1968; Batterman & Patel, 1968). However, none of those authors were able to obtain absolute values of structure factors to better than 1%, even on favourable materials such as silicon. In the present measurements on the 220 Bragg reflexion from silicon, we have obtained values of structure factors with deviations of less than 0·1% between separate experiments. Since this is the highest precision ever claimed for a structure factor measurement, we will describe the experimental technique in some detail.

Such precision of course finds immediate applications in studies of the electron distribution in crystals, in discussions of the relative merits of the various theoretical models by which atomic scattering factors are calculated and in the evaluation of possible sources of systematic error in the more conventional techniques by which structure factors are measured. In this context it may be important to notice that the Pendellösung method involves only the coherent part of the X-ray scattering amplitude.

Theory

The spherical wave theory of diffraction by highly perfect crystals has been thoroughly developed in a series of papers by Kato (1960, 1961*a*,*b*, 1968*a*,*b*). In addition, the influence on Pendellösung fringes of crystal imperfections, including elastic strains, has been investigated both theoretically (Kato, 1964) and experimentally (Hart, 1966) in considerable detail.

In the symmetric Laue case, with which we are exclusively concerned, the intensity field I_h of the Bragg reflected waves in the spherical wave case of a section pattern (Kato & Lang, 1959) is

$$I_h=A\pi^2\Delta_0^{-2}\operatorname{cosec}^2\theta\{J_0(\pi\varrho\Delta_0^{-1})\}^2\,, \qquad (1)$$

where A is a constant and θ is the Bragg angle. J_0 is the zero order Bessel function and ϱ is the depth in the crystal measured along the net plane from the X-ray entrance surface (we are concerned only with

the intensity field along the net plane). Δ_0 is given by

$$\Delta_0 = \frac{\lambda \cos \theta}{C(\chi_h \chi_{\bar{h}})^{\frac{1}{2}}} \quad (2)$$

where λ is the X-ray wavelength and C is the polarization factor which is 1 for σ-case polarization and $|\cos 2\theta|$ for the π-case polarization. For the low order Bragg reflexions from silicon with Mo $K\alpha_1$ and Ag $K\alpha_1$ radiations we can write, with sufficient accuracy,

$$(\chi_h \chi_{\bar{h}})^{\frac{1}{2}} = \frac{r_e \lambda^2 |F_h|}{\pi V} . \quad (3)$$

r_e is the classical electron radius and F_h is the structure factor for the h-order Bragg reflexion from the unit cell whose volume is V.

For those fringes which are not too close to the entrance surface of the crystal we may use the asymptotic form of the Bessel function so that

$$I_h = 2\pi A \Delta_0^{-1} \varrho^{-1} \operatorname{cosec}^2\theta \sin^2 \left(\pi \varrho \Delta_0^{-1} + \frac{\pi}{4}\right) . \quad (4)$$

That this equation is essentially correct has been demonstrated experimentally by Homma, Ando & Kato (1966) and by the present authors (1968).

Unpolarized X-ray sources

In our experiments we use a normal electron-excited X-ray tube so that we assume that the incident beam is essentially unpolarized. Explicitly including both principal polarization components, equation (4) can be developed as

$$I_h = A' \left\{ \sin^2\pi \left(\frac{\varrho}{\Delta_0^\sigma} + \tfrac{1}{4}\right) + |\cos 2\theta| \sin^2\pi \left(\frac{\varrho}{\Delta_0^\pi} + \tfrac{1}{4}\right) \right\} . \quad (5)$$

Δ_0^σ and Δ_0^π are the values of Δ_0 obtained from equation (2) with $C=1$ and $C=|\cos 2\theta|$ respectively. Further, it is conceptually convenient to work in terms of $\bar{\Delta}_0$ the polarization-averaged value of Δ_0, corresponding to $C=\frac{1}{2}(1+|\cos 2\theta|)$. We can define *interference orders* on the exit surface by

$$t = n_0^\sigma \Delta_0^\sigma = n_0^\pi \Delta_0^\pi = \bar{n}_0 \bar{\Delta}_0 , \quad (6)$$

where t is the crystal thickness. Consider now the test function

$$I \propto \sin^2\pi \left(\frac{\varrho}{\bar{\Delta}_0} + \tfrac{1}{4}\right) . \quad (7)$$

Its minima occur when the term in brackets is equal to an integer m. On the other hand, it is easily shown that extrema of I_h occur when $(\varrho/\bar{\Delta}_0 + \frac{1}{4}) = m + \delta m$ where δm is calculated from equation (8) below:

$$\tan (2\pi\delta m) = \tan [2\pi(m-\tfrac{1}{4}) \tan^2\theta] \frac{\cos^2 2\theta - 1}{\cos^2 2\theta + 1} . \quad (8)$$

The fringe shift δm is zero if $\frac{1}{2}q \cot^2\theta = (m-\frac{1}{4})$ or, more familiarly, if

$$(m-\tfrac{1}{4}) = \tfrac{1}{2}q(1+|\cos 2\theta|)/(1-|\cos 2\theta|) = qN$$

where q is an integer (Hart, 1963; Hart & Lang, 1965; Hattori, Kuriyama & Kato, 1965). The minima of I_h occur near the fringe orders defined by the following scheme:

$$0 < \bar{n}_0 < \tfrac{1}{2}N \quad \text{minima near } \bar{n}_0 = l - \tfrac{1}{4}$$

$$\tfrac{1}{2}(2k-1)N < \bar{n}_0 < \tfrac{1}{2}(2k+1)N \begin{cases} \text{minima near } \bar{n}_0 = l + \tfrac{1}{4} \\ \text{if } k \text{ is odd} \\ \text{minima near } \bar{n}_0 = l - \tfrac{1}{4} \\ \text{if } k \text{ is even ,} \end{cases} \quad (9)$$

and l is an integer.

The influence of absorption

The theory so far has not included absorption. Kato (1968*a*) has recently published the full spherical wave theory of Pendellösung fringes in section patterns showing that small fringe shifts do occur if absorption is present. For *minima* the fractional change in fringe position $\Delta m/m$ is given by:

$$\begin{aligned} \Delta m/m = & [\pi(m-\tfrac{1}{4})]^{-2}\{\kappa_0\pi(m-\tfrac{1}{4}) \sinh^2[\kappa\pi(m-\tfrac{1}{4})] \\ & + \tfrac{1}{2} \sinh^2[\kappa\pi(m-\tfrac{1}{4})] \\ & - \tfrac{1}{2}\kappa\pi(m-\tfrac{1}{4}) \sinh [2\kappa\pi(m-\tfrac{1}{4})]\} . \end{aligned} \quad (10)$$

The terms κ and κ_0 are essentially the ratios of imaginary to real parts of scattering factors. They are defined in Kato's paper referred to above. In the present experiments κ and κ_0 are approximately 1×10^{-2} and 6×10^{-3} for Mo $K\alpha_1$ and Ag $K\alpha_1$ respectively, so that these shifts are completely negligible.

Elastic deformation

Potentially the most serious error which may arise in Pendellösung fringe measurements of X-ray structure factors is that due to elastic strain. It is particularly insidious because homogeneous deformations can change the positions of fringes without altering their shapes (see for example Hart, 1966, Fig. 5). For small deformations in which the strain gradients are functions of the depth ϱ in the crystal and do not vary rapidly within planes of constant ϱ, the interference order n and the local fringe spacing Δ are given by (Hart, 1966; Kato, 1964):

$$n = n_0(1 + \tfrac{1}{6}p^2) \quad (11)$$

$$\Delta = \Delta_0(1+p^2)^{-\frac{1}{2}} \quad (12)$$

where

$$p = \frac{St \tan \theta}{C\lambda^2 |F_h|} \quad (13)$$

and S is determined by the strain field. These equations are strictly correct when the strain gradient is constant. For a particular low-angle Bragg reflexion, we can write approximately

$$p \propto S \frac{t}{\lambda} . \quad (14)$$

Equations (11) to (13) can be written in terms of

polarization averaged parameters with sufficient accuracy for the present purpose.

Sample preparation

We have attempted to eliminate the parasitic fringe shifts mentioned in the previous section since, in principle, it is better to eliminate corrections than to be forced to make them.

Shifts due to polarization

To avoid fringe shifts caused by the overlap of the patterns for the two states of X-ray polarization we work only with crystal thicknesses near $t_q = qN\bar{\Delta}_0$. For fringe shifts $\delta m < 0{\cdot}01$ we may use only Pendellösung fringes with orders $qN - 4 \le \bar{n}_0 \le qN + 4$ for Mo $K\alpha_1$ radiation and $qN - 11 \le \bar{n}_0 \le qN + 11$ for Ag $K\alpha_1$ radiation (equation 8). For the 220 Bragg reflexion of silicon, N is approximately 14 for Mo $K\alpha_1$ and 23 for Ag $K\alpha_1$ radiations.

It is interesting to notice that, for small Bragg angles, $t_q \propto q\lambda^{-3}$ so that the set of thicknesses t_q for Ag $K\alpha_1$ radiation coincide almost exactly with the even members of the set of t_q values for Mo $K\alpha_1$ radiation. Thus, on one sample we can take measurements under optimum conditions with two different X-ray wavelengths.

Accordingly, a stepped wedge shaped specimen was prepared, the wedge angle being chosen so that each step contained approximately four fringes spaced roughly 1 mm apart on the wedge. The mean thicknesses of the two steps were chosen near $q = 1$ and $q = 2$ for the 220 reflexion of Ag $K\alpha_1$ radiation. To avoid geometrical errors the $(\bar{1}11)$ entrance surface of the wedge was normal to the 220 Bragg planes to within 1 min arc and the wedge edge was within 5 min arc of the $[1\bar{1}2]$ direction.

Elastic deformations

By double crystal topography (Bonse & Kappler, 1958; Bonse, 1961; Hart, 1968) we have found growth banding in the low oxygen float-zone dislocation-free silicon which we have been using for structure factor measurements. The bands are normal to the $[\bar{1}11]$ growth axis of the crystal and have a spatial periodicity of approximately 100 μm with lattice parameter fluctuations $\Delta d/d \simeq 1 - 2 \times 10^{-7}$. This elastic strain is harmless in the dynamical sense ($p = 0$ in equations 11 and 12) for all symmetric Laue-case reflexions in the $[\bar{1}11]$ zone. Consequently we have chosen to use the 220 Bragg reflexion and not any of the other available 220 type reflexions from symmetry-related lattice planes. It is particularly advantageous if $(\bar{1}11)$ is chosen as the entrance surface.

Experimental method

We have used the method of section patterns (Kato & Lang, 1959) employing a ribbon beam of X-rays 10 μm wide with a small vertical divergence. The source to crystal distance was 75 cm. The wedge-shaped crystal was mounted on a slide so that section patterns could be obtained at known positions in the specimen with respect to a reference edge. Pendellösung fringes were photographed on Ilford type L4 nuclear emulsion plates and at each observation position patterns were obtained both with Mo $K\alpha_1$ and with Ag $K\alpha_1$ radiations.

After the X-ray experiments were completed, the wedge was cut parallel to the Bragg planes at the places where section patterns had been obtained and the crystal thickness was directly measured with a travelling microscope. The positions of Pendellösung minima were measured both with a microdensitometer on the original plates and by eye estimation on high contrast photographic enlargements of the original plates. There was no detectable systematic difference between the results obtained by the two methods. A complete collection of results is given in Table 1.

From the local fringe spacing on the wedges and the measured crystal thickness, an approximate fringe order $\bar{n}_0^*$ was obtained by division for each Pendellösung minimum. Then the exact fringe orders $\bar{n}_0$ were assigned, following the scheme of equation (9). The deviation $|\bar{n}_0 - \bar{n}_0^*|$ was usually less than 0·2 and was never greater than 0·3. The Pendellösung periodicity $\bar{\Delta}_0$ was then calculated from $\bar{\Delta}_0 = t/\bar{n}_0$, equation (6). Since the only source of random error is in the thick-

Table 1. *Measured crystal thickness t* μm *and assigned fringe orders* $\bar{n}_0$ *in the Pendellösung fringe patterns at three separate positions A, B and C in the specimen*

$\bar{n}_0$	A		B		C		$\bar{n}_0$	A		B		C	
Mo $K\alpha_1$	t	$\bar{\Delta}_0$	t	$\bar{\Delta}_0$	t	$\bar{\Delta}_0$	Ag $K\alpha_1$	t	$\bar{\Delta}_0$	t	$\bar{\Delta}_0$	t	$\bar{\Delta}_0$
25·75	973	37·786	973	37·786	968	37·592	20·25	968	47·802	970	47·901	967	47·753
26·75	1011	37·794	1013	37·869	1011	37·794	21·25	1012	47·624	1016	47·812	1013	47·671
27·75	1047	37·730	1050	37·838	1048	37·766	22·25	1062	47·730	1067	47·955	1060	47·640
28·75	1085	37·739	1088	37·843	1086	37·774	23·25	1110	47·742	1115	47·957	1111	47·785
29·75	1123	37·748	1123	37·748	1124	37·782							
Mean	37·760 ± 0·024		37·817 ± 0·040		37·742 ± 0·060		Mean	47·725 ± 0·050		47·906 ± 0·050		47·712 ± 0·057	
52·75	1992	37·763	1999	37·896	1992	37·763	41·75	1994	47·760	2001	47·928	1993	47·737
53·75	2030	37·767	2033	37·823	2029	37·749	42·75	2037	47·649	2042	47·766	2040	47·719
54·75	2068	37·772	2070	37·808	2068	37·772	43·75	2087	47·703	2084	47·634	2089	47·749
55·75	2109	37·830	2103	37·722	2106	37·776							
Mean	37·783 ± 0·023		37·812 ± 0·047		37·765 ± 0·009		Mean	47·704 ± 0·028		47·776 ± 0·101		47·735 ± 0·011	

ness calibration, we have weighted the results giving double weight to the thick crystal measurements so that

$$\bar{\Delta}_0 = 37{\cdot}78 \pm 0{\cdot}04\ \mu\text{m for Mo } K\alpha_1$$
$$\bar{\Delta}_0 = 47{\cdot}75 \pm 0{\cdot}07\ \mu\text{m for Ag } K\alpha_1 .$$

These correspond to:

$$f = 8{\cdot}478 \pm 0{\cdot}008 \text{ for Mo } K\alpha_1$$
$$f = 8{\cdot}448 \pm 0{\cdot}012 \text{ for Ag } K\alpha_1 .$$

Both results are *experimental values with no corrections for thermal motion or anomalous dispersion*, at 20 °C. The errors quoted here are weighted mean deviations from the weighted mean. As far as we can tell from the rather limited number of readings, a histogram of the results in Table 1 appears to represent a normal distribution.

Without using the thickness measurements, we can calculate the ratio of assigned fringe orders for the two wavelengths used at particular points in the section patterns. These are collected together in Table 2. The ratios were calculated at the minima of the Mo $K\alpha_1$ patterns, the corresponding order of the Ag $K\alpha_1$ patterns being calculated by linear interpolation between minima. Each ratio is the mean from four positions in the thin wedge or from three measurements in the thick wedge.

Table 2. *Ratio of assigned fringe orders at particular points in the crystal*

Each thin wedge value is the mean of four measurements and each thick wedge value is the mean of three measurements.

Experiment	*A*	*B*	*C*
Thin wedge	1·2643	1·2651	1·2639
Thick wedge	1·2625	1·2636	1·2645
Weighted mean ratio	1·2640 ± 0·0009		

The order ratio is a function only of the wavelengths, Bragg angles and atomic scattering factors for the two radiations. If the scattering factor is independent of wavelength the order ratio should be 1·2596 (from equations 2 and 6). We must therefore conclude that the difference between this ratio and those in Table 2 is significant and that, at 20 °C

$$f(\text{Mo } K\alpha_1)/f(\text{Ag } K\alpha_1) = 1{\cdot}0035 \pm 0{\cdot}0007 .$$

Residual strains

In spite of the precautions taken one might be tempted to suggest that residual strains could be responsible for this surprising result.

However, close inspection of Table 1 and Table 2 reveals that there is no systematic variation of either $\bar{\Delta}_0$ or of the order ratio with crystal thickness. We can also obtain measurements of $\bar{\Delta}_0$ by subtraction of the thin crystal results from the thick crystal results in Table 1. This process, for Mo $K\alpha_1$ radiation, gives $\bar{\Delta}_0 = 37{\cdot}84 \pm 0{\cdot}07\ \mu$m, in excellent agreement with the other values already noted.

Theoretically (equation 11) $\bar{\Delta}_0$ should decrease systematically with increasing thickness and the order ratios too should change systematically if the crystal is strained. We therefore must conclude that these crystals are effectively perfect.

Discussion

It is, in some cases, not possible to compare our results with other authors because they have made corrections for anomalous dispersion and/or thermal effects without explicitly stating the correction used. An attempt to compare results is given below:

(1) Hattori *et al.* (1965): $f = 8{\cdot}58 \pm 0{\cdot}09$ 'maximum deviation'. The radiation was Mo $K\alpha_1$ and/or Ag $K\alpha_1$.

(2) Hart (1966): $f = 8{\cdot}52 \pm 0{\cdot}04$ mean deviation. Ag $K\alpha_1$ radiation. The original paper contains a systematic error since fringe orders were assigned on the plane wave theory. Correction to the spherical wave theory (equation 9) yields the above result.

(3) De Marco & Weiss (1965): $f_0 = 8{\cdot}70 \pm 0{\cdot}06$ whence ,if $\Delta f' = 0{\cdot}07$ and $e^{-M} = 0{\cdot}973$ we calculate: $f = 8{\cdot}53 \pm 0{\cdot}06$ at 20 °C.

(4) Göttlicher & Wölfel (1959): $f = 8{\cdot}48 \pm 0{\cdot}02$.

(5) Kato & Tanemura (1967): $f = 8{\cdot}45$. This paper contained a systematic error in that the dispersion correction was omitted from the refractive index calculation. The above result assumes $\Delta f' = 0{\cdot}07$.

It is interesting that the present measurements lie approximately 0·6% below the mean of all the previous measurements. We have not succeeded in finding any adequate explanation for the discrepancy though this may become apparent as more high precision measurements of scattering factors become available.

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DISCUSSION

WEISS (TO HART): (*a*) These results are most impressive – it would be excellent if they are indeed as accurate as claimed. We would then have a valuable reference standard.

(*b*) There is one point and that was the relatively large difference between Mo and Ag measurements. Did you consider dispersion corrections?

HART: Yes, the total correction is ~0·4% but the difference is still 0·3%. We have good internal consistency in all measurements. So far we cannot explain this difference. It is the only embarrassing feature.

BATTERMAN (TO KATO): Referring to the table of Debye temperatures where you compare Professor Borrmann's and your own results, you said that Borrmann's measurements were consistent with 296° at room temperature and 330° at 20°K. On the other hand, Professor Borrmann in his talk gave a value of 290° and this appeared to fit over the whole temperature range considered. Have you any comments?

KATO: We do not know the true value of the Debye temperature so I have used the room temperature value derived from our own Pendellösung measurements. Your theoretical values are slightly higher than these and it is possible that the difference between the room and low-temperature values quoted may account for the difference between our value and the theoretical at room temperature.

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Determination of the Atom Form Factor by High Voltage Electron Diffraction

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A new method of determining the atom form factors from the values of accelerating voltages for which the second order Kikuchi line disappears is applied to aluminum, iron, nickel and copper. The accuracy of the method is briefly discussed and the numerical results of the atom form factors are given.

It was reported recently that the second order Kikuchi line in electron diffraction disappears at a certain accelerating voltage E_c due to the many-beam dynamical interaction combined with the relativistic change of electron mass (Watanabe, Uyeda & Kogiso, 1968). The present authors used the effect for determining the Fourier coefficient of the crystal potential for the first order V_1. Thus the corresponding value of the X-ray atom form factor f_1^x was determined also (Watanabe, Uyeda & Fukuhara, 1968). In the present note, an experimental procedure is described and the error in f_1^x obtained by this method is discussed. The numerical results for aluminum, iron, nickel and copper are given.

Thin foils were obtained by electropolishing from 99·9 to 99·99% metal plates annealed in vacuum, and examined with a 500 kV electron microscope. Diffraction patterns were taken from areas of a few microns diameter or smaller by the selected area diffraction technique at various accelerating voltages. The accelerating voltage of each diffraction pattern was determined from the analysis of the Kikuchi pattern with an accuracy of 1% (Uyeda, Nonoyama & Kogiso, 1965). The value of E_c was determined with an accuracy of 10 kV by examining a series of diffraction patterns taken at various accelerating voltages. The values of E_c were measured for the 220 reflexion of iron and the 222 reflexion of aluminum, nickel and copper (Table 1). The X-ray atom form factors f_1^x for the first order reflexion were determined using the measured values of E_c and known values of f_n^x for the second and higher order reflexions ($n=2, 3, \ldots$).

The percentage error in f_1^x thus obtained can be roughly estimated on the basis of the Bethe's second