X-RAY LINE-BREADTH ANALYSIS OF GROWTH AND DEFORMATION FAULTING IN HEXAGONAL CLOSE-PACKED STRUCTURES

BY P. RAMA RAO AND T. R. ANANTHARAMAN

(Department of Metallurgy, Banaras Hindu University, Varanasi-5)

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1. INTRODUCTION

The occurrence of stacking faults in a close-packed structure was first postulated by Edwards and Lipson and Wilson to explain the anomalous X-ray line broadening in hexagonal close-packed (h.c.p) cobalt obtained by spontaneous transformation of face-centred cubic (f.c.c) cobalt on cooling from above 420°C. Interest in detection and estimation of stacking faults in both f.c.c and h.c.p structures has increased in recent years following Barrett's suggestion that such structural irregularities may be introduced in metallic structures during either phase transformation or plastic deformation. Most of the recent work concerning measurement and interpretation of faulting, however, has been confined to f.c.c metals and alloys, chiefly because of the comparative simplicity of the method of estimation for cubic structures. Although very few h.c.p structures have been examined, there is clear evidence for profuse and complex faulting in many of them.

Debye-Scherrer patterns of faulted structures obtained as diffractometer records or photometer traces have been utilised for evaluating fault parameters from variation in line breadths or by Fourier analysis of line shapes. Spreadborough has compared results obtained by both these methods and tentatively concluded that either method may be employed for studies of growth faulting, but the Fourier method is more reliable in case of heavy deformation faulting. Apart from the fact that the Fourier method involves time-consuming computations, it cannot be claimed that an unequivocal interpretation of the Fourier coefficients is always possible, especially in cases of mixed faulting. Moreover, the Fourier method does not permit easy corrections for serious limitations in the measuring range as observed in actual practice. The analysis of line-breadths is relatively simpler and has the merit of being readily applicable to a large number of reflections in a powder pattern.
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The aim of this paper is to develop X-ray line-breadth analysis as a reasonably accurate and easily applicable method for studies of even a mixed distribution of stacking faults in an h.c.p. structure. Possible simplifications in the methods of calculation have been explored and a reliable procedure arrived at. The suggested method is illustrated with reference to Wilson's results, the earliest for h.c.p. cobalt, and for two other samples of pure cobalt annealed at 500 and 800° C. respectively.

II. Notation

Miller-Bravais indices ... HKIL
Lattice parameters ... a, c
Density of growth and deformation stacking faults respectively ... a, a'
Integral and half-peak breadths respectively in reciprocal space along L direction of any HKIL-reflection ... β, β̅
Experimentally observed integral and half-peak breadths in reciprocal space along L direction ... β', β̅'
Integral and half-peak breadths of reflections with odd and even L values respectively ... β₀, βₑ, β̅₀, β̅ₑ
Integral breadths of reflections with odd and even L values due to growth and deformation faults respectively ... β₀₉, βₑ₉, β₀ₑ, βₑₑ
Half-peak breadths of reflections with odd and even L values due to growth and deformation faults respectively ... β̅₀₉, β̅ₑ₉, β̅₀ₑ, β̅ₑₑ
Integral and half-peak diffraction breadths in radians for any HKIL-reflection ... β₀, βₑ, β̅₀, β̅ₑ
Distance from peak along L direction in reciprocal space for any HKIL-reflection ... ν
Wavelength of X-radiation ... λ
Bragg angle ... θ
Diffracted intensity at distance ν from peak in reciprocal space ... I, K, k₁ to k₉

Constants

...
III. THEORY OF X-RAY DIFFRACTION

If the normal stacking sequence for the close-packed planes of an h.c.p. lattice is indicated as...ABABAB..., the growth and deformation stacking fault contributing to \( a \) and \( a' \) may respectively be indicated by breaks in the regular sequence such as...ABAB/CBCB... and...ABAB/CACA... A growth or deformation fault can thus be visualised as an insertion of three or four layers respectively of the regular f.c.c. sequence...ABCABC...in an h.c.p. lattice. Faulting can be mixed with either type predominating.

Following earlier workers, the effects of a random distribution of faults on the intensity distribution in reciprocal lattice for an h.c.p. structure can be expressed as

\[
I_\nu = \frac{K (1 - \rho^2)}{1 - 2\rho \cos \pi \nu + \rho^2}
\]

(1)

where

\[
\rho = 0 \text{ for reflections with } H - K = 3n,
\]

\[
= -\frac{a}{2} + \frac{1}{2} \{a^2 + (4 - 8a)(1 - 3a' + 3a'^2)\}\]

for reflections with \( H - K \neq 3n \) and even \( L \) value

and

\[
= \frac{a}{2} + \frac{1}{2} \{a^2 + (4 - 8a)(1 - 3a' + 3a'^2)\}\]

for reflections with \( H - K \neq 3n \) and odd \( L \) value.

The values of \( \beta, \bar{\beta}, \beta_{2\theta}, \bar{\beta}_{2\theta} \) may be arrived at from equation (1) and the symmetry of the h.c.p. lattice as

\[
\beta = \frac{2(1 - \rho)}{(1 + \rho)} \quad \beta_{2\theta} = \frac{\lambda^2 L \beta}{c^2 \cdot \sin 2\theta}
\]

\[
\bar{\beta} = \frac{2}{\pi} \cos^{-1} \left( \frac{4\rho - \rho^2 - 1}{2\rho} \right) \quad \bar{\beta}_{2\theta} = \frac{\lambda^2 L \bar{\beta}}{c^2 \cdot \sin 2\theta}
\]

(2)

It is theoretically possible to arrive at \( a \) and \( a' \) from the experimentally observed values of \( \beta_{2\theta} \) or \( \bar{\beta}_{2\theta} \) from equations (2), but the expressions for \( \rho \) need simplification to render the calculations practically feasible. Moreover, equation (1) implies an intensity distribution extending throughout the \( L \) direction in reciprocal space. A suitable correction is essential since the
measuring range by experiment is generally less than a quarter \( (\nu < 0.25) \) of the full range \( (\nu = \pm 1) \) underlying equations (2).

### IV. SUGGESTED APPROXIMATIONS

Wilson has simplified the expression for \( \rho \) for small values of \( \alpha \) as

\[
\rho = 1 - \frac{3}{2} \alpha \quad \text{for reflections with } H - K \neq 3n \text{ and } L \text{ even}
\]

and

\[
\rho = 1 - \frac{1}{2} \alpha \quad \text{for reflections with } H - K \neq 3n \text{ and } L \text{ odd}.
\]

A similar simplification can be made in the case of \( \alpha' \) to lead to the following relations for the integral breadths:

\[
\beta_e^0 = 3 \beta_0^0 = \frac{3\alpha}{2}
\]

\[
\beta_e^n = \beta_0^n = \frac{3\alpha'}{2}.
\]

Table I shows the effect of this approximation for \( \alpha \) values in the range 0.01–0.20. It is clear that the errors introduced are rather high for heavy deformation faulting as has been observed for h.c.p. cobalt, titanium and cobalt-nickel alloys. Less drastic approximation leads to

**Table 1**

*Actual and approximated integral breadths in reciprocal space of X-ray reflections from faulted h.c.p. structures*

<table>
<thead>
<tr>
<th>( \alpha ) or ( \alpha' )</th>
<th>( \beta_e^0 ) (Actual)</th>
<th>( \beta_e^0 ) (Wilson's approximation)</th>
<th>( \beta_e^0 ) (Authors' approximation)</th>
<th>( \beta_e^0 ) (Actual)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>0.0152</td>
<td>0.0150</td>
<td>0.0151</td>
<td>0.0151</td>
</tr>
<tr>
<td>0.02</td>
<td>0.0306</td>
<td>0.0300</td>
<td>0.0305</td>
<td>0.0304</td>
</tr>
<tr>
<td>0.04</td>
<td>0.0625</td>
<td>0.0600</td>
<td>0.0619</td>
<td>0.0612</td>
</tr>
<tr>
<td>0.06</td>
<td>0.0958</td>
<td>0.0900</td>
<td>0.0942</td>
<td>0.0926</td>
</tr>
<tr>
<td>0.08</td>
<td>0.1306</td>
<td>0.1200</td>
<td>0.1276</td>
<td>0.1246</td>
</tr>
<tr>
<td>0.10</td>
<td>0.1675</td>
<td>0.1500</td>
<td>0.1622</td>
<td>0.1570</td>
</tr>
<tr>
<td>0.15</td>
<td>0.2663</td>
<td>0.2250</td>
<td>0.2535</td>
<td>0.2398</td>
</tr>
<tr>
<td>0.20</td>
<td>0.3795</td>
<td>0.3000</td>
<td>0.3530</td>
<td>0.3240</td>
</tr>
</tbody>
</table>
\[
\beta_e^G = 3\beta_0^G = \frac{6\alpha}{4 - 3\alpha}
\]
\[
\beta_e^D = \beta_0^D = \frac{6\alpha'}{4 - 3\alpha'}.
\]
(4)

The values of the last column of Table I show that this approximation leads to considerably less errors when high stacking fault values are measured.

Warren\textsuperscript{11} has recently suggested an approximation for calculations using half-peak breadths, which can be expressed for each type of faulting as:

\[
\bar{\beta}_e^G = 3\bar{\beta}_0^G = \frac{3\alpha}{\pi}
\]
\[
\bar{\beta}_e^D = \bar{\beta}_0^D = \frac{3\alpha'}{\pi}.
\]
(5)

Table II brings out the effect of this approximation and shows that the following relations are to be preferred:

\[
\bar{\beta}_e^G = 3\bar{\beta}_0^G = \frac{4\alpha}{4 - 3\alpha}
\]
\[
\bar{\beta}_e^D = \bar{\beta}_0^D = \alpha'.
\]
(6)

**Table II**

*Actual and approximated half-peak breadths in reciprocal space of X-ray reflections from faulted h.c.p. structures*

<table>
<thead>
<tr>
<th>(\alpha) or (\alpha')</th>
<th>(\bar{\beta}_e^G) (Actual)</th>
<th>(\bar{\beta}_0^G) (Warren's approximation)</th>
<th>(\bar{\beta}_e^G) (Authors' approximation)</th>
<th>(\bar{\beta}_e^D) (Authors' approximation)</th>
<th>(\bar{\beta}_0^D) (Actual)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>0.0096</td>
<td>0.0099</td>
<td>0.0101</td>
<td>0.0100</td>
<td>0.0096</td>
</tr>
<tr>
<td>0.02</td>
<td>0.0194</td>
<td>0.0197</td>
<td>0.0203</td>
<td>0.0200</td>
<td>0.0193</td>
</tr>
<tr>
<td>0.04</td>
<td>0.0399</td>
<td>0.0395</td>
<td>0.0412</td>
<td>0.0400</td>
<td>0.0390</td>
</tr>
<tr>
<td>0.06</td>
<td>0.0611</td>
<td>0.0592</td>
<td>0.0628</td>
<td>0.0600</td>
<td>0.0589</td>
</tr>
<tr>
<td>0.08</td>
<td>0.0834</td>
<td>0.0790</td>
<td>0.0851</td>
<td>0.0800</td>
<td>0.0796</td>
</tr>
<tr>
<td>0.10</td>
<td>0.1069</td>
<td>0.0987</td>
<td>0.1081</td>
<td>0.1000</td>
<td>0.1004</td>
</tr>
<tr>
<td>0.15</td>
<td>0.1715</td>
<td>0.1481</td>
<td>0.1690</td>
<td>0.1500</td>
<td>0.1542</td>
</tr>
<tr>
<td>0.20</td>
<td>0.2476</td>
<td>0.1974</td>
<td>0.2353</td>
<td>0.2000</td>
<td>0.2100</td>
</tr>
</tbody>
</table>
When both types of stacking faults coexist in an h.c.p. lattice, the above approximations may be synthesised as:

(a) Wilson’s approximation for integral breadths:
\[ \beta_0 = \frac{a}{2} + \frac{3}{2} a' \]
\[ \beta_e = \frac{3}{2} a + \frac{3}{2} a' \]

(b) Suggested new approximation for integral breadths:
\[ \beta_0 = \frac{2a + 6a'}{4 - 3a - 3a'} \]
\[ \beta_e = \frac{6a + 6a'}{4 - 3a - 3a'} \]  \hspace{1cm} (7)

(c) Warren’s approximation for half-peak breadths:
\[ \beta_0 = \frac{a + 3a'}{\pi} \]
\[ \beta_e = \frac{3a + 3a'}{\pi} \]

(d) Suggested new approximation for half-peak breadths:
\[ \bar{\beta}_0 = \frac{4a + 12a'}{3 (4 - 3a)} \]
\[ \bar{\beta}_e = \frac{4a + 4a'}{4 - 3a} \]  \hspace{1cm} (8)

Equations (7) and (8) may thus be chosen for determining densities of stacking faults in an h.c.p. structure provided data for at least one right pair of reflections are available. The limitations of the actual measuring range for the intensity distribution are not taken into account in this determination. The error introduced by ignoring the necessary correction for the same will be appreciable if \( a \) and \( a' \) are greater than about 0.02.

V. CORRECTION FOR LIMITATIONS IN MEASURING RANGE

Starting from equation (1), an exact correction for limitations in measuring the intensity distribution can be worked out. As shown by Wilson, the observed integral breadth is
\[ \beta' = \frac{4}{\pi} \arctan \left( \frac{1 + \rho}{1 - \rho} \tan \frac{\pi \nu}{2} \right) - \frac{2 \nu (1 - \rho^2)}{1 - 2 \rho \cos \pi \nu + \rho^2}. \]  \hspace{1cm} (9)
A similar derivation for half-peak breadths gives

$$\bar{\beta}' = \frac{2}{\pi} \cos^{-1} \left\{ \frac{1 + \rho^2 + \cos \pi \nu (1 + \rho^2 - 4\rho)}{2 - 2\rho (1 + \cos \pi \nu - \rho)} \right\}. \quad (10)$$

Tables III–VI bring out the impact of limitations in the measuring range on the observed breadth values. The errors in ignoring equations (9) and (10) are generally larger for integral breadths than for half-peak breadths. The corrections for reflections with odd L values are considerably lower than for those with even L values.

**TABLE III**

*Dependence of integral breadth on measuring range for h.c.p. structures with growth faults*

<table>
<thead>
<tr>
<th>$\nu$</th>
<th>$a = 0.01$</th>
<th>$a = 0.05$</th>
<th>$a = 0.10$</th>
<th>$a = 0.15$</th>
<th>$a = 0.20$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\beta_0^{g}$</td>
<td>$\beta_e^{g}$</td>
<td>$\beta_0^{e}$</td>
<td>$\beta_e^{e}$</td>
<td>$\beta_0^{g}$</td>
</tr>
<tr>
<td>0.025</td>
<td>0.0047</td>
<td>0.0119</td>
<td>0.0176</td>
<td>0.0286</td>
<td>0.0257</td>
</tr>
<tr>
<td>0.050</td>
<td>0.0049</td>
<td>0.0134</td>
<td>0.0214</td>
<td>0.0443</td>
<td>0.0365</td>
</tr>
<tr>
<td>0.100</td>
<td>0.0049</td>
<td>0.0143</td>
<td>0.0236</td>
<td>0.0582</td>
<td>0.0447</td>
</tr>
<tr>
<td>0.150</td>
<td>0.0050</td>
<td>0.0145</td>
<td>0.0245</td>
<td>0.0642</td>
<td>0.0481</td>
</tr>
<tr>
<td>0.200</td>
<td>0.0050</td>
<td>0.0147</td>
<td>0.0250</td>
<td>0.0675</td>
<td>0.0498</td>
</tr>
<tr>
<td>0.300</td>
<td>0.0050</td>
<td>0.0148</td>
<td>0.0254</td>
<td>0.0711</td>
<td>0.0517</td>
</tr>
<tr>
<td>0.500</td>
<td>0.0051</td>
<td>0.0149</td>
<td>0.0258</td>
<td>0.0741</td>
<td>0.0532</td>
</tr>
<tr>
<td>1.000</td>
<td>0.0051</td>
<td>0.0150</td>
<td>0.0261</td>
<td>0.0760</td>
<td>0.0542</td>
</tr>
<tr>
<td>Ideal</td>
<td>0.0051</td>
<td>0.0152</td>
<td>0.0263</td>
<td>0.0790</td>
<td>0.0558</td>
</tr>
</tbody>
</table>

The data in Tables III–VI can be graphically reproduced to extrapolate the actual breadth from observed values for any value of $\nu$. For any measured range $\nu$, plots of $a$ or $a'$ and relevant correction factors can also be made to obtain the required factors for $a$ and $a'$ calculated from equations (7) or (8). These can be substituted in the following equations to get more accurate values of $a$ and $a'$:

$$\beta_0 = k_1 a + k_5 a'; \quad \bar{\beta}_0 = k_3 a + k_6 a'$$

$$\beta_e = k_2 a + k_6 a'; \quad \bar{\beta}_e = k_4 a + k_4 a'. \quad (11)$$
### TABLE IV
Dependence of half-peak width on measuring range for h.c.p. structures with growth faults

<table>
<thead>
<tr>
<th>( \nu )</th>
<th>( \alpha = 0.01 )</th>
<th>( \alpha = 0.05 )</th>
<th>( \alpha = 0.10 )</th>
<th>( \alpha = 0.15 )</th>
<th>( \alpha = 0.20 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \tilde{\beta}_{o}^{G} )</td>
<td>0.0029</td>
<td>0.0092</td>
<td>0.0151</td>
<td>0.0289</td>
<td>0.0251</td>
</tr>
<tr>
<td>( \tilde{\beta}_{e}^{G} )</td>
<td>0.0029</td>
<td>0.0095</td>
<td>0.0163</td>
<td>0.0410</td>
<td>0.0317</td>
</tr>
<tr>
<td>( \tilde{\beta}_{o}^{C} )</td>
<td>0.0029</td>
<td>0.0095</td>
<td>0.0166</td>
<td>0.0474</td>
<td>0.0344</td>
</tr>
<tr>
<td>( \tilde{\beta}_{e}^{C} )</td>
<td>0.0029</td>
<td>0.0096</td>
<td>0.0167</td>
<td>0.0490</td>
<td>0.0350</td>
</tr>
<tr>
<td>( \beta_{o}^{G} )</td>
<td>0.0029</td>
<td>0.0096</td>
<td>0.0167</td>
<td>0.0495</td>
<td>0.0352</td>
</tr>
<tr>
<td>( \beta_{e}^{G} )</td>
<td>0.0029</td>
<td>0.0096</td>
<td>0.0168</td>
<td>0.0500</td>
<td>0.0354</td>
</tr>
<tr>
<td>( \beta_{o}^{C} )</td>
<td>0.0029</td>
<td>0.0096</td>
<td>0.0168</td>
<td>0.0502</td>
<td>0.0354</td>
</tr>
<tr>
<td>( \beta_{e}^{C} )</td>
<td>0.0029</td>
<td>0.0096</td>
<td>0.0168</td>
<td>0.0503</td>
<td>0.0355</td>
</tr>
<tr>
<td>Ideal</td>
<td>0.0029</td>
<td>0.0096</td>
<td>0.0168</td>
<td>0.0503</td>
<td>0.0355</td>
</tr>
</tbody>
</table>

### TABLE V
Dependence of integral breadth on measuring range for h.c.p. structures with deformation faults

<table>
<thead>
<tr>
<th>( \nu )</th>
<th>( \alpha' = 0.01 )</th>
<th>( \alpha' = 0.05 )</th>
<th>( \alpha' = 0.10 )</th>
<th>( \alpha' = 0.15 )</th>
<th>( \alpha' = 0.20 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0119</td>
<td>0.0284</td>
<td>0.0318</td>
<td>0.0326</td>
<td>0.0329</td>
</tr>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0134</td>
<td>0.0437</td>
<td>0.0571</td>
<td>0.0618</td>
<td>0.0639</td>
</tr>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0142</td>
<td>0.0570</td>
<td>0.0885</td>
<td>0.1056</td>
<td>0.1153</td>
</tr>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0145</td>
<td>0.0628</td>
<td>0.1056</td>
<td>0.1341</td>
<td>0.1529</td>
</tr>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0146</td>
<td>0.0660</td>
<td>0.1160</td>
<td>0.1532</td>
<td>0.1802</td>
</tr>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0148</td>
<td>0.0694</td>
<td>0.1280</td>
<td>0.1766</td>
<td>0.2161</td>
</tr>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0149</td>
<td>0.0722</td>
<td>0.1386</td>
<td>0.1989</td>
<td>0.2527</td>
</tr>
<tr>
<td>( \beta_{e}^{p} )</td>
<td>0.0150</td>
<td>0.0740</td>
<td>0.1456</td>
<td>0.2142</td>
<td>0.2789</td>
</tr>
<tr>
<td>Ideal</td>
<td>0.0151</td>
<td>0.0768</td>
<td>0.1570</td>
<td>0.2398</td>
<td>0.3240</td>
</tr>
</tbody>
</table>
VI. APPLICATION TO TRANSFORMED H.C.P. COBALT

The data in Wilson's classic paper on stacking faults in transformed h.c.p. cobalt powder may now be explained more satisfactorily on the basis of the above treatment. As already recognised, the unsatisfactory agreement (Table VII) between the fault parameter values from different reflections is mostly due to the earlier assumption that the specimen contained only growth stacking faults. Revised on the basis of equations (7), the observed and calculated \( \beta \) values show much better agreement and also confirm later estimations\(^4,6\) using Fourier analysis of line shapes.

Table VIII gives our results for pure cobalt powder (99.98% pure) vacuum annealed in two batches for a day at 500 and 800\(^{\circ}\) C. respectively. The pure diffraction breadth \( \beta' \) has been obtained from the integral breadths, \( B \) and \( b \), of reflections affected and unaffected by faulting respectively employing the relation:

\[
\beta' = B - \frac{b^e}{B}
\]
that has already been used by earlier workers. No such simple relation has been suggested for half-peak breadths. A further justification for using this empirical equation is that good agreement has been observed over a wide range of \( \frac{b}{B} \) values between integral breadth values obtained from this equation and by Fourier analysis. A similar procedure has been used to arrive at \( \nu \) from observed measuring ranges of broadened and unbroadened reflections. The actual values of \( \alpha \) and \( \alpha' \) are consistent with those earlier obtained by Fourier analysis for pure cobalt specimens annealed at different temperatures. This result as also the low percentage mean deviations between observed and calculated \( \beta \) for these two specimens may be taken as confirmation of the soundness of the suggested line-breadth analysis method for the study of faulting in h.c.p. structures. It has, however, to be stressed that the mean deviations may not be so low generally as in this case and a deviation of 5-10% may be considered satisfactory.
TABLE VIII

Evaluation of stacking fault densities in transformed h.c.p. cobalt powder
(Crystal-reflected Ni Kα radiation: Camera radius 45 mm.)

<table>
<thead>
<tr>
<th>HKIL</th>
<th>Annealed at 500°C</th>
<th>Annealed at 800°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>β'(Calculated for</td>
<td>β'(Calculated for</td>
</tr>
<tr>
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Mean deviation between calculated and observed β

- Annealed at 500°C: 3.8%
- Annealed at 800°C: 2.5%

VII. SUMMARY

Approximations employed so far in the evaluation of growth and deformation faulting in h.c.p. structures from integral and half-peak X-ray line breadths have been critically examined. An improved and yet simple procedure has been suggested to study faulting, mixed or otherwise, in h.c.p. structures. Necessary corrections for limitations of the measuring range have been worked out for integral and half-peak breadths over a wide range of growth and deformation fault parameters. The suggested procedure is illustrated by application to a study of faulting in spontaneously transformed cobalt powder.

REFERENCES


