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Instrumental Corrections for X-ray Reflexions from Thin Monocrystalline Films

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Correction curves for instrumental broadening are given for the cases that the physical broadening profile is $(\sin^2 Kx)/(Kx)^2$ and the instrumental profile is either $(1+\kappa^2x^2)^{-1}$ or $(1+\kappa^2x^2)^{-2}$ or $\exp\{-\kappa^2x^2\}$.

The interpretation of recent diffractometer measurements of thin monocrystals requires the observed reflexions to be corrected for instrumental influences. Only then is it possible to compare the thickness calculated from the breadth of the reflexions with that determined from fringe spacings, electron microscopy, gravimetry, optical interferrometry *etc.* It is customary and convenient to obtain the instrumental broadening from a standard specimen which is known to be free from physical broadening. For thin films there is apparently the complication that the standard specimen has to be thicker in order to be free from size broadening. The penetration of the X-ray beam into the specimen then causes an increase of the instrumental broadening for the thicker film. However, the total width of the aberration profile due to penetration is in most cases so small that it and its changes with specimen thickness can be neglected. An estimate can be obtained by assuming that the crystal reflects as a delta function. Calculating the penetration into the specimen as function of the deviation ε ($2\varepsilon=2\theta$-diffractometer angle; Wilson, 1963), the unnormalized penetration profile becomes

$p(\varepsilon)=0$ for $\varepsilon<0$
$p(\varepsilon)=\exp\{-4\mu R(\sin\varepsilon)/\sin 2\theta\}$ for $0\le\varepsilon\le t(\cos\theta)/R$
$p(\varepsilon)=0$ for $\varepsilon>t(\cos\theta)/R$

where R is the diffractometer radius and t the thickness of the specimen. Thence the total breadth, $2\varepsilon_e$, of the penetration profile is determined either by the thickness of the specimen $2\varepsilon_e=2t(\cos\theta)/R$ or, in the case of thick samples, by the exponential decrease of the profile $2\varepsilon_e=2{\cdot}30\,(\sin 2\theta)/\mu R$ for $p(\varepsilon_e)/p(0)=0{\cdot}01$. For the 200 Au reflexion (Cu radiation, $\mu=4350\ \text{cm}^{-1}$, $R=17$ cm) the maximum possible width is only $2\varepsilon_e=0{\cdot}00125\,^{\circ}2\theta$, which occurs for thicknesses greater than 2×10^{-4} cm. Wilson (1950, 1963) has calculated the penetration and the flat-specimen plus penetration profile for the more complicated case of a powder specimen.

For the standard crystal the compounding of several aberrations often gives an instrumental profile $b(x)$ which can be approximated by one of the functions (a), (b) or (c) given in Table 1. The diffraction profile for a thin crystal is $\sin^2(pB\varepsilon)/\sin^2(B\varepsilon)$, where p is the number of diffracting planes with spacing a and $B=2\pi a$ $(\cos\theta)/\lambda$ (James, 1958). A good approximation is $\beta(x)$ $(\cos\theta)/\lambda$ (James, 1958). A good approximation is $\beta(x)=(\sin^2Kx)/(Kx)^2$ which has the same integral breadth and area as the original function. The diffraction fringes predicted by the above function have been observed and can be used to determine p (Croce, Devant, Gandais & Marrud, 1962; Vook & Witt, 1965).

The observed profile is the convolution $B(x)=\int\beta(u)b(x-u)du$ (Jones, 1938) which has the solution $B(x)=T^{-1}[T[B(x)]T[b(x)]]$ where the transforms are defined as $T^{\pm1}[f(x)]=\int_{-\infty}^{+\infty}f(x)\exp\{\pm2\pi iux\}dx$. The functions, their integral breadths and the transforms are given in Table 1.

Case (d) is included here without application in the present context. The results for $B(x)$, for the integral breadth B and for the ratio of the breadths b/B are as follows.

(a) $B(x)=(\pi/K^2)[(K+\frac{1}{2}\kappa-\frac{1}{2}\kappa C_xe^-)(1+\kappa^2x^2)^{-1}$
$\quad-(\kappa+x\kappa^2S_xe^--\kappa C_xe^-)(1+\kappa^2x^2)^{-2}]$
$B=(\pi K/\kappa)(K-\frac{1}{2}\kappa+\frac{1}{2}\kappa e^-)^{-1}$
$b/B=1-\frac{1}{2}(\beta/b)+\frac{1}{2}(\beta/b)\exp\{-2b/\beta\}$

(b) $B(x)=(\pi/4K)[(\kappa/K)-2C_xe^--(\kappa/K)C_xe^-](1$
$\quad+\kappa^2x^2)^{-1}+(\pi/K)[1+(\kappa/K)-(\kappa/K)C_xe^-$
$\quad+C_xe^--x\kappa S_xe^-](1+\kappa^2x^2)^{-2}-(2\pi\kappa/K^2)$
$\quad[x\kappa S_xe^--C_xe^-+1](1+\kappa^2x^2)^{-3}$
$B=(\pi/\kappa)[2-(3\kappa/2K)+e^-+(3\kappa/2K)e^-]^{-1}$
$b/B=1-(3\beta/8b)+\frac{1}{2}\exp\{-4b/\beta\}$
$\quad+(3\beta/8b)\exp\{-4b/\beta\}$

(c) $B(0)=(2\pi/K^2)[K\,\text{erf}\{\sqrt{2}K/\kappa\}$
$\quad-(\kappa/2\sqrt{\pi})(1-\exp\{-K^2/\kappa^2\})]$
$B=(\pi^{1\cdot5}/\kappa K)[B(0)]^{-1}$
$b/B=2\,\text{erf}\{\sqrt{2\pi}b/\beta\}-(\beta/\pi b)(1-\exp\{-\pi b^2/\beta^2\})$

(d) $B(x)=(\pi/2K^2\kappa^2)[(K-\kappa)C_xx^{-2}+(K+\kappa)x^{-2}-S_xx^{-3}]$
$B=\pi\kappa/(K\kappa-\frac{1}{3}K^2)\qquad K<\kappa$
$b/B=(b/\beta)-\frac{1}{3}b^2/\beta^2\qquad \beta>b$

where $C_x=\cos 2Kx$, $S_x=\sin 2Kx$, $e^-=\exp\{-2K/\kappa\}$ and $\text{erf}(x)=(2\pi)^{-\frac{1}{2}}\int_0^x\exp\{-y^2/2\}dy$.

For case (c), $B(x)$ could not be obtained; instead $B(0)$ and B were calculated following Jones (1938). If in case (d) $b>\beta$ and $\kappa<K$ the relevant results are obtained by interchanging K with κ, and β with b. The correction graphs β/B *versus* b/B are shown in Fig. 1.

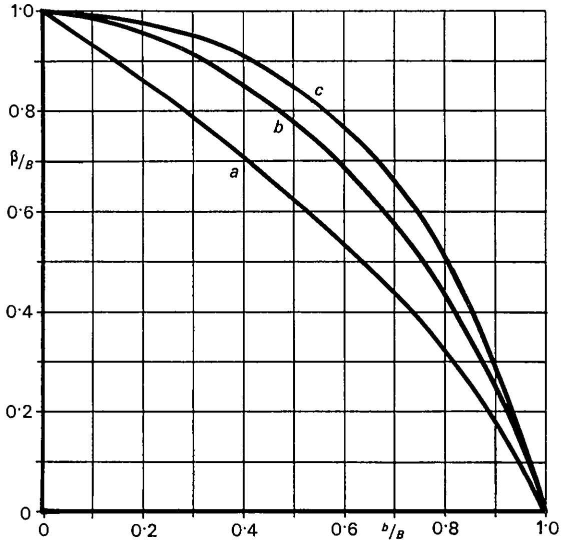

Fig. 1. Correction curves for size profile $\beta(x)=(\sin^2Kx)/(Kx)^2$ and instrumental profiles (a): $b(x)=(1+\kappa^2x^2)^{-1}$; ($b$): $b(x)=(1+\kappa^2x^2)^{-2}$; ($c$): $b(x)=\exp\{-\kappa^2/x^2\}$.

Table 1. *Breadths and transforms of the profiles*

Profile	Breadth	Transform
$\beta(x)=(\sin^2Kx)/(Kx)^2$	$\beta=\pi/K$	$T[\beta(x)]=\pi(K-\pi u)/K^2;\ u\le K/\pi$ $=0;\ u>K/\pi$
(a) $b(x)=(1+\kappa^2x^2)^{-1}$	$b=\pi/\kappa$	$T[b(x)]=(\pi/\kappa)\exp\{-2\pi\lvert u\rvert/\kappa\}$
(b) $b(x)=(1+\kappa^2x^2)^{-2}$	$b=\pi/2\kappa$	$T[b(x)]=(\pi/2\kappa^2)(\kappa+2\pi u)\exp\{-2\pi u/\kappa\}$
(c) $b(x)=\exp\{-\kappa^2x^2\}$	$b=\sqrt{\pi}/\kappa$	$T[b(x)]=(\sqrt{\pi}/\kappa)\exp\{-\pi^2u^2/\kappa^2\}$
(d) $b(x)=(\sin^2\kappa x)/(\kappa x)^2$	$b=\pi/\kappa$	$T[b(x)]=\pi(\kappa-\pi u)/\kappa^2;\ u\le\kappa/\pi$ $=0;\ u>\kappa/\pi$

Jones (1938) did a calculation similar to the present case (*b*), using numerical integration methods and an experimental profile for $b(x)$ which approximates closely to $(1+\kappa^2x^2)^{-2}$.

In the above analysis it is assumed that all experimental profiles have been corrected for α_1–α_2 overlap, and that influences which vary slowly with 2θ are negligible or have been taken care of.

The results from line broadening and from fringe spacing should be identical provided the specimen is not faulted and is free of strains normal to the diffracting planes. The ease with which measurements of broadening and fringe spacing can be performed, allows a quick test for strains and faults in the specimens.

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The Crystal Structure of Bis-(*N*-ethylsalicylaldiminato)copper(II)

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The structure of the complex bis-(*N*-ethylsalicylaldiminato)copper(II) of molecular formula $C_{18}H_{20}N_2O_2Cu$ has been determined with two-dimensional Weissenberg and precession data by the heavy atom method and refined with three-dimensional Weissenberg data. The crystals are monoclinic with $a=9{\cdot}824$, $b=8{\cdot}432$, $c=20{\cdot}989$ Å, $\beta=101°$. The space group is $P2_1/c$ with four molecules per unit cell. The coordination polyhedron around the copper atom is a distorted tetrahedron. The angle between the two coordination planes is 35° 52′ and the step angles between the coordination planes and the planes of the salicyl chelates are 11° 30′ and 22° 44′. No interaction is possible between copper atoms.

Introduction

The α form of bis-(*N*-methylsalicylaldiminato)copper(II), the structure of which has been determined by Lingafelter, Simmons, Morosin, Scheringer & Freiburg (1961), shows a planar arrangement of the ligand atoms about the copper atom. The space groups of the normal *N*-substituted salicylaldiminatocopper(II) complexes (Frasson & Panattoni, 1962), indicate that in the *N*-n-butyl and *N*-n-pentyl derivatives the coordination configuration of the copper atom is planar. In fact the presence of two copper atoms in a cell of space group $P2_1/c$ requires that they be at centres of symmetry. For the *N*-ethyl and *N*-n-propyl derivatives, on the basis of space group considerations alone, no information is available on the type of arrangement of ligands around the copper atom. We have now carried out the determination of the structure of bis-(*N*-ethylsalicylaldiminato)copper(II).

Experimental

The compound, of formula $C_{18}H_{20}N_2O_2Cu$, was prepared by the method of Charles (1957) as modified by Sacconi, Ciampolini, Maggio & Del Re (1960). Black crystals, which have the form of thick plates, were obtained.

The crystal data are as follows:

Molecular weight 354·9
$a=\ 9{\cdot}824\pm0{\cdot}012$ Å
$b=\ 8{\cdot}432\pm0{\cdot}012$
$c=20{\cdot}989\pm0{\cdot}026$
$\beta=101°\pm10'$
$V=1706$ Å^3
$F(000)=748$
$\varrho_m=1{\cdot}38$ g.cm^{-3}
$\varrho_{\text{calc}}=1{\cdot}40$ g.cm^{-3}
$Z=4$.

The systematic absence of reflexions $h0l$ for l odd and $0k0$ for k odd uniquely determined the space group as $P2_1/c$. The cell parameters were measured from precession photographs and the density was measured by flotation. A very small fragment (mean dimensions: 0·015 cm) was selected for intensity measurements. Equi-inclination Weissenberg photographs of $h0l$ through $h6l$ and of $0kl$ were obtained with nickel-filtered Cu $K\alpha$ radiation on a Wiebenga camera with integration in one direction only (Scatturin, Panattoni,