J. Appl. Cryst. (1988). **21**, 363–368

X-ray Diffraction Line Broadening Due to Dislocations in Non-Cubic Materials. II. The Case of Elastic Anisotropy Applied to Hexagonal Crystals

By Radomír Kužel Jr

Department of Semiconductor Physics, Faculty of Mathematics and Physics, Charles University, 121 16 Prague 2, Czechoslovakia

and Peter Klimanek

Section of Metallurgy and Materials Technology, Academy of Mining, 9200 Freiberg, German Democratic Republic

(*Received* 9 *November* 1987; *accepted* 18 *March* 1988)

Abstract

The calculation of orientation factors determining the anisotropy of dislocation-induced broadening of X-ray diffraction lines in crystals is treated for elastically anisotropic materials in terms of a general formalism. The application of the procedure is demonstrated by representation of a computer program and numerical calculations of orientation factors for different slip systems in hexagonal polycrystals with randomly oriented grains. A comparison of the results with those obtained in the approximation of elastic isotropy shows that the anisotropy of the diffraction-line broadening is essentially caused by the geometrical part of the orientation factor. In most cases the elastic anisotropy of the crystal leads only to small corrections.

1. Introduction

According to the theoretical work of Krivoglaz (1967, 1983) and his co-workers and Wilkens (1970), the broadening of X-ray diffraction peaks due to dislocations in crystalline materials is essentially determined by the quantity

$$B(\mathbf{m}) = (Qb)^2(8\pi)^{-1}\rho\chi(\mathbf{m}) \qquad (1)$$

where Q is the magnitude of the diffraction vector, $\mathbf{Q} = (4\pi/\lambda)(\mathbf{s} - \mathbf{s}_0)$, with direction $\mathbf{m} = \mathbf{Q}/Q$, b is the magnitude of the Burgers vector and ρ is the total density of dislocations. The so-called orientation factor $\chi(\mathbf{m})$ is determined by the orientation $\mathbf{m}$ of the diffraction vector with respect to the Burgers vector $\mathbf{b}$ of the dislocations. It is associated with a given type $\langle uvw\rangle\{hkl\}$ of slip systems and depends on the lattice geometry, the elastic behaviour of the irradiated material, and the dislocation type.

In paper I (Klimanek & Kužel, 1988) a general procedure for the calculation of the factors $\chi = \chi(\mathbf{m})$ occurring in X-ray diffraction of polycrystals with randomly oriented grains was presented and applied to hexagonal materials in the approximation of elastic isotropy. In such conditions the orientation factors could be described by analytical expressions.

In the present paper the factors are calculated without the presumptions of elastic isotropy. After general considerations the case of hexagonal crystals is again treated. For some special dislocations it is also possible to derive analytical formulae for the orientation factors (Raychenko & Martynova, 1970), but the calculations and also the resulting expressions are rather cumbersome. Numerical methods are straightforward here because they permit one to take into account all types of dislocations in a given material. For this reason a computer program, *HEXAN*, was developed and applied to the important slip systems of hexagonal crystals. The results are compared with those obtained from the approximation of elastic isotropy, and some conclusions concerning the practical use of X-ray profile analysis for the characterization of the dislocation content in hexagonal polycrystals are drawn.

2. General treatment of orientation factors for elastically anisotropic materials

According to paper I the orientation factors related to a special type of dislocation (slip systems) in polycrystals can always be written in the form

$$\chi_p = \sum_{K,L} \langle G_{KL}\rangle E_{KL} \qquad K, L = 1, 2, \ldots, 6. \qquad (2)$$

The components of this expression have the following meaning. The matrix G is the so-called geometrical part of the orientation factor. It can be obtained by calculation of the directional cosines of the angles between the diffraction vector $\mathbf{Q}$ and the axes of an appropriate slip coordinate system and subsequent averaging over all symmetrically equivalent slip systems. The matrix E is the so-called elastic part of the

0021-8898/88/040363-06$03.00

orientation factor. Its components are defined by the integrals (*cf.* paper I)

$$\hat{E}_{ijkl} = (1/\pi) \int_0^{2\pi} D_{ij} D_{kl} \, \mathrm{d}\varphi \quad i,k = 1,2,3, \; j,l = 1,2, \quad (3)$$

where the quantities D_{ij} are proportional to the derivatives $\partial u_i/\partial x_j$ of the displacement field of the dislocation and φ is a polar angle. The matrix $\hat{E}$ can be rewritten into the matrix E by means of the rule: $K = i$ for $j = 1$, $K = i + 3$ for $j = 2$ and $L = k$ for $l = 1$, $L = k + 3$ for $l = 2$ (for example).

In the calculations of the orientation factors χ of elastically anisotropic materials the matrix G is obtained in the same manner as in the approximation of elastic isotropy (*cf.* paper I), while for the determination of the matrix components $\hat{E}_{ijkl}$ the general form of the displacement field around an isolated dislocation in an elastically anisotropic medium must be used (Steeds, 1973; Teodosiu, 1982). In general, the computation of the orientation factors has to be performed numerically. For this purpose it is very convenient to use the theory of Lekhnitskiy as briefly described in the book of Teodosiu (1982). Such an approach has two main merits: applicability to the general case (any crystallographic axis system) and convenience for numerical calculations. This paper describes, however, only what is necessary for the calculation of orientation factors.

The displacement field of a dislocation should be expressed in Cartesian coordinates where the displacement vector does not depend on one particular coordinate (*e.g.* x_3). For this reason a special slip coordinate system is introduced (Fig. 1 of paper I). It is a system with the axis x_3 parallel to the dislocation line and x_2 perpendicular to the slip plane. Other coordinate systems, of course, can be used as well. The component u_k of the displacement vector can be expressed then by the formula

$$u_k = (1/\pi) \, \mathrm{Im} \left(\sum_{\alpha=1}^{3} A_{k\alpha} D_\alpha \ln z_\alpha \right) \quad (4)$$

where $z_\alpha = x_1 + p_\alpha x_2$, $\mathrm{Im}\, p_\alpha > 0$, is a complex variable. The coefficients p must be determined by solution of the following sectic equation arising from the condition of equilibrium (Steeds, 1973; Teodosiu, 1982):

$$l(p) = l_2(p) l_4(p) - l_3(p) = 0 \quad (5)$$

with

$$l_2(p) = S_{55}p^2 - 2S_{45}p + S_{44}$$

$$l_3(p) = S_{15}p^3 - (S_{14} + S_{56})p^2 + (S_{25} + S_{46})p - S_{24}$$

$$l_4(p) = S_{11}p^4 - 2S_{16}p^3 + (2S_{12} + S_{66})p^2 - S_{26}p + S_{22}.$$

The quantities S_{ij} are the so-called reduced elastic compliances which are defined in terms of the normal elastic compliances s_{ij} by the relation

$$S_{ij} = s_{ij} - (s_{3i}s_{3j}/s_{33}). \quad (6)$$

The sectic equation (5) can be solved numerically for any symmetry. In this connection it is often possible to reduce it to a bicubic equation by the choice of a convenient coordinate system (Steeds, 1973). In such a case another transformation of s_{ij} has to be performed. The analytical solution of (5) then becomes simple. In general it has three complex conjugate pairs of roots. They can be arranged according to the conditions

$$l_2(p_1) = 0 \quad l_2(p_2) = 0 \quad l_4(p_4) = 0.$$

Then the coefficients $A_{k\alpha}, D_\alpha$ in (4) can be calculated through S_{ij}, p and b_k (components of the Burgers vector). The corresponding relations are given in the book of Teodosiu (1982), chapter 10. Now, with the aid of (5), (4) and (3) one is able to get the elements of the matrix E, and the orientation factors can be calculated through (2) as described by Klimanek & Kužel (1988).

3. Calculation of orientation factors for hexagonal materials

3.1. *The computer program HEXAN*

The computer program *HEXAN* has been written in Fortran for the calculation of orientation factors in hexagonal polycrystalline materials (extent 750 lines). It takes advantage of the procedure described above and includes the most important dislocation types of hexagonal materials (Predvoditelev & Troickyi, 1973). The program *HEXAN* is interactive and also available for common personal computers. It is described briefly and schematically below.

(1) Input: elastic stiffness constants c_{ij}, lattice parameters a, c, Burgers vectors **b**, slip planes type $(hkil)$, dislocation character ϕ.

(2) The calculation of the elastic part E of the orientation factor includes the following steps: compliances s'_{ij}; local compliances s_{ij} (Steeds, 1973); reduced compliances S_{ij}; coefficients p_α of the bicubic equation; complex coefficients A, D in the relation for the displacement field (4); and integration (3) using a modified Simpson method.

(3) The calculation of the geometrical part G includes the following procedures: transformation of the crystallographic coordinate system into the so-called physical orthonormal coordinate system used in the theory of elasticity (Steeds, 1973; Teodosiu, 1982); input of the diffraction vector **Q** in terms of Miller indices; calculation of the matrix elements G_{ijkl} for all the crystallographically equivalent slip systems $\langle uvwz \rangle \{hkil\}$; and averaging of the matrix components G_{KL}.

(4) Output: output of the χ_p value for a given reflection.

(5) Input: choice of the new program path.

Table 1. *Degrees of elastic anisotropy for selected hexagonal materials*

The ratios of lattice parameters a/c are also shown.

Material	A	A_c^*	A_s^*	a/c	Reference
Group *A*					
Magnesium	0·98	0·01	0·34	0·616	(2)
Titanium	1·33	0·005	1·74	0·622	(1)
Tungsten carbide	1·41	0·96	1·43	1·024	(3)
Group *B*					
Zinc	0·59	9·88	13·7	0·539	(1)
Cadmium	0·53	9·41	9·06	0·53	(1)
Thallium	2·64	0·15	15·1	0·625	(2)

References for the elastic stiffness constants:
(1) Landolt-Börnstein (1966).
(2) Chung & Buessem (1968).
(3) Lee & Gilmore (1982).

Table 2. *Influence of elastic anisotropy on the elements of matrix E demonstrated on magnesium (low anisotropy) and zinc (high anisotropy)*

Dislocation type	Magnesium						Zinc					
Edge (isotropic)	1·72	0	0	0	−0·45	0	1·75	0	0	0	−0·31	0
		1·72	0	−2·39	0	0		1·75	0	−2·46	0	0
			0	0	0	0			0	0	0	0
				6·06	0	0				5·88	0	0
					0·38	0					0·34	0
						0						0
Anisotropic $\langle 2\bar{1}\bar{1}0\rangle$ $\{0001\}$	1·62	0	0	0	−0·39	0	1·44	0	0	0	−0·34	0
		1·6	0	−2·27	0	0		2·35	0	−3·26	0	0
			0	0	0	0			0	0	0	0
				6·35	0	0				7·19	0	0
					0·31	0					0·75	0
						0						0
Screw (isotropic)	Only non-zero elements						$E_{33} = E_{66} = \pi = 3{\cdot}141592$					
Anisotropic $\langle 2\bar{1}\bar{1}0\rangle$	$E_{33} = 3{\cdot}099$ $E_{66} = 3{\cdot}185$						$E_{33} = 2{\cdot}415$ $E_{66} = 4{\cdot}086$					

For application of the program to crystals with other than hexagonal symmetry two modifications must be introduced. Firstly, the sectic equation (5) must be solved numerically because generally it cannot be reduced to a bicubic one. Secondly, the procedure for the calculation of the matrix elements G_{KL} of crystallographically equivalent slip systems must be created for each symmetry separately.

3.2. *Results of numerical calculations*

Numerical calculations of orientation factors have been carried out for the materials listed in Table 1. The substances can roughly be divided into two groups; (*a*) crystals with a low degree of elastic anisotropy (*e.g.* Mg, Ti); and (*b*) crystals with a high degree of elastic anisotropy (*e.g.* Zn, Cd). In order to characterize the degree of elastic anisotropy, which cannot be done so unambiguously as in the case of cubic materials, the following relations were used (Table 1):

$$A = 2(s_{11} - s_{12})/s_{44} \tag{7}$$

and

$$\begin{aligned} A^*_{\text{compression}} &\equiv A_c^* = (K_V - K_R)/(K_V + K_R) \\ A^*_{\text{shear}} &\equiv A_s^* = (G_V - G_R)/(G_V + G_R). \end{aligned} \tag{8}$$

In accordance with (7) (Steeds, 1973) the case of elastic isotropy is described by $A = 1$ as for cubic crystals. Nevertheless, as indicated by Table 1, a more instructive characterization of the elastic behaviour of hexagonal materials is possible by means of (8) (Chung & Buessem, 1968), where K and G are the averaged bulk and shear moduli, respectively, and the subscripts V and R refer to the well known Voigt and Reuss averaging procedures. Elastic isotropy is indicated by $A^* = 0$ in this case. For a comprehensive characterization of the elastic anisotropy of hexagonal materials it is necessary, in principle, to use both quantities A_c^* and A_s^*.

In order to demonstrate the influence of the elastic anisotropy on the orientation factors for both groups of materials described above, the matrices E of magnesium and zinc as calculated by *HEXAN* and those obtained in the approximation of elastic isotropy are compared in Table 2. In agreement with the factors A of Table 1 the results for magnesium (group A) are very similar, while in the case of zinc (group B) some elements E_{KL} significantly differ.

The orientation factors resulting from these matrices for the most important slip systems of hexagonal materials (Predvoditelev & Troickyi, 1973; see also paper I) are plotted in Fig. 1 as functions $\chi = \chi_p(\delta)$ of the angle between the reciprocal-lattice vector associated with the operating diffraction vector Q and the c axis of the lattice cell. The dashed lines represent the orientation factors obtained in the approximation of elastic isotropy (*cf.* paper I). In this connection it must be noticed that for edge dislocations of the type $\langle 2\bar{1}\bar{1}0\rangle\ \{0\bar{1}0\}$ and for screw dislocations with Burgers vectors $\langle 0001\rangle$ the displacements are equal in elastically isotropic and anisotropic media and cannot be calculated by the procedure described here, which fails for the case of elastic isotropy (Steeds, 1973).

The orientation factors for Mg and Zn can be considered as typical for the groups of crystal structures distinguished above. The results for the other materials of Table 1 have been deposited.* The dependence of the orientation factors on the dislo-

*Results for titanium, tungsten carbide, cadmium and thallium have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44860 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

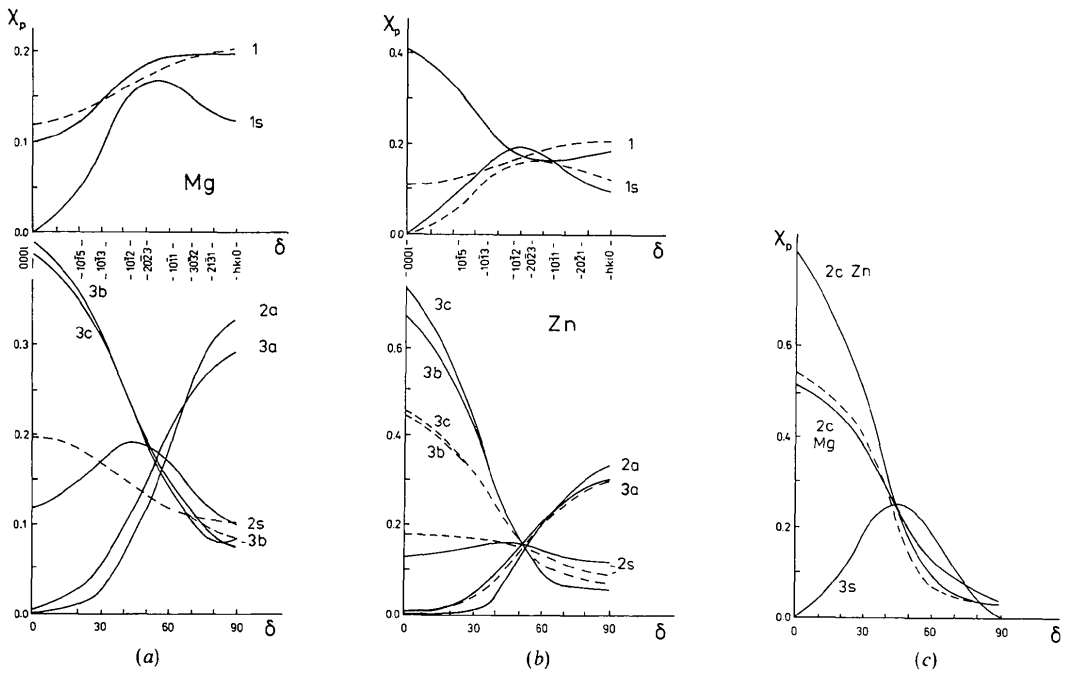

Fig. 1. Dependence of orientation factors on the angle between the reciprocal-lattice vector connected with the operating diffraction vector and the c axis of the hexagonal lattice. Calculations were carried out for various dislocations (slip systems) in (*a*) magnesium and (*b*) zinc regardless of the actual probability of their presence in these materials.

The notation is as follows (Burgers vectors parallel to the given directions):

screw dislocations	1*s* $\langle 2\bar{1}\bar{1}0\rangle$, 2*s* $\langle 11\bar{2}3\rangle$
	3*s* $\langle 0001\rangle$
edge dislocations	1 basal $\langle 2\bar{1}\bar{1}0\rangle$
	2*a* prismatic $\langle 2\bar{1}\bar{1}0\rangle$
	2*b* prismatic $\langle 11\bar{2}3\rangle$
	2*c* prismatic $\langle 0001\rangle$
	3*a* pyramidal $\langle 2\bar{1}\bar{1}3\rangle\ \{01\bar{1}1\}$
	3*b* pyramidal $\langle 2\bar{1}\bar{1}3\rangle\ \{10\bar{1}1\}$
	3*c* pyramidal $\langle 2\bar{1}\bar{1}3\rangle\ \{2\bar{1}\bar{1}2\}$.

Indices of some reflections are also shown.

In (*c*) the results for less-common Burgers vector are drawn. The dashed line corresponding to the calculations in the approximation of elastic isotropy is the same for zinc and magnesium.

cation character ϕ is represented for both cases of elastic isotropy and anisotropy and for several X-ray reflections in Fig. 2. In practice, especially for polycrystals, the presence of more slip-system types must be considered and average orientation factors should be calculated. An illustrative example is shown in Fig. 3 for zinc. The average factors $\langle\chi_p\rangle$ were calculated by means of the formula

$$\langle\chi_p\rangle = c_1 b_1^2 \chi_{p1} + (1 - c_1) b_2^2 \chi_{p2}. \qquad (9)$$

χ_{p1}, χ_{p2} are average orientation factors for dislocations with Burgers vectors $a/3\ \langle 2\bar{1}\bar{1}0\rangle$ and $a/3\ \langle 11\bar{2}3\rangle$, respectively, c is the fraction of the dislocation density corresponding to the former type of the Burgers vector, and b is the magnitude of this Burgers vector. The factors were calculated under the same assumptions as in paper I.*

The results obtained in such a way should again be typical for hexagonal materials because it is the change of the fraction c which has the most significant influence on the resulting orientation factor.

One note must be given here. In X-ray diffraction experiments the information for each reflection hkl is taken only from the properly oriented grains (*e.g.* parallel to the sample surface in conventional diffractometry). The orientation factors given above were calculated under the assumption that slip systems are equally activated in all grains irrespective of their orientation. In particular, this was applied in the calculations of the matrix G (all the symmetrically equivalent slip systems) and derivations of the curves in Figs. 1–3 (equal dislocation density in all grains). Generally an additional weighting must be introduced, but this is not the subject of this paper.

*These assumptions are: (*a*) there are 50% of screw and 50% of edge dislocations; (*b*) the concentrations of edge dislocations with the Burgers vector of the first kind in the slip planes {0001}, {01$\bar{1}$0}, {01$\bar{1}$1} are the same; and (*c*) the concentration of edge dislocations with the Burgers vector of the second kind in the slip planes {01$\bar{1}$1} and {11$\bar{2}$2} are the same.

3.3. *Discussion of the results*

From the numerical calculations of the factors χ a number of conclusions concerning the influence of the elastic anisotropy on the X-ray diffraction line broadening of hexagonal crystals can be drawn.

(i) Provided pure screw and edge dislocations are considered, the differences between the orientation factors obtained by the general procedure of the paper and those calculated from the approximation of elastic isotropy are important only for edge dislocations on the basal planes {0001} and screw dislocations with Burgers vectors $a/3\ \langle 11\bar{2}3\rangle$. In all other cases the effect of the elastic anisotropy can practically be neglected. In particular this is true for the materials belonging to group *A* of Table 1 (*i.e.* for a low degree of elastic anisotropy). For the substances of group *B* (high degree of elastic anisotropy) the differences are greater in general (notice the different scales of Figs. 1*a* and *b*), but again unimportant with respect to the possible uncertainties of experimentally determined line widths and Fourier coefficients.

(ii) For mixed dislocations, as follows from Fig. 2, the large differences between the orientation factors

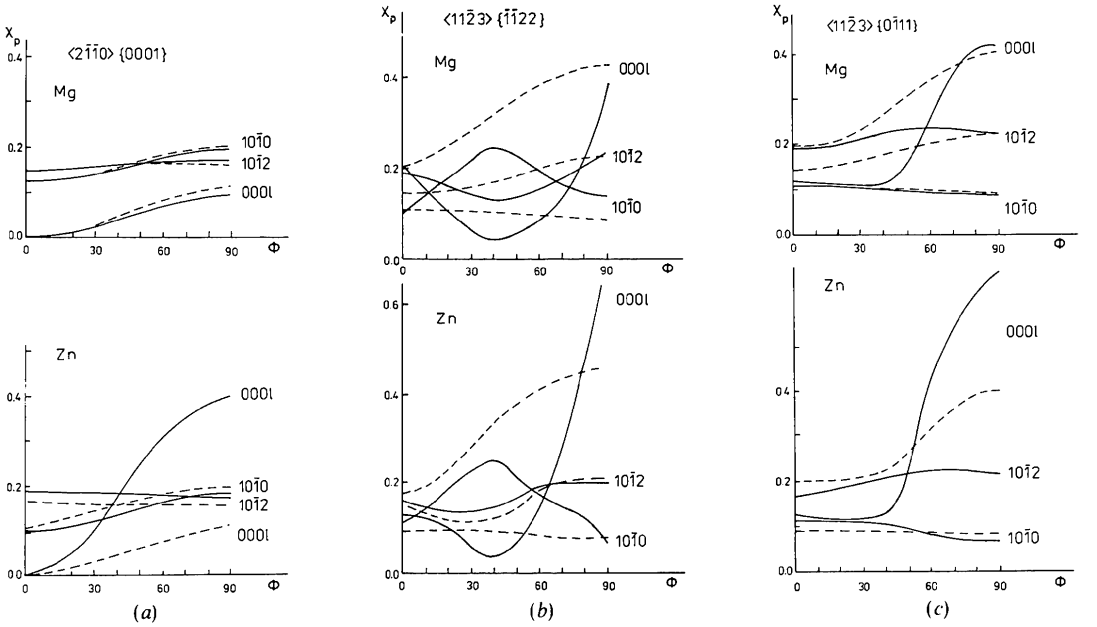

Fig. 2. Dependence of orientation factors χ_p on the dislocation character ϕ for magnesium and zinc, various reflections and slip systems: (*a*) basal, (*b*) pyramidal $\langle 11\bar{2}3\rangle$ $\{\bar{1}\bar{1}22\}$, (*c*) pyramidal $\langle 11\bar{2}3\rangle$ $\{0\bar{1}11\}$.

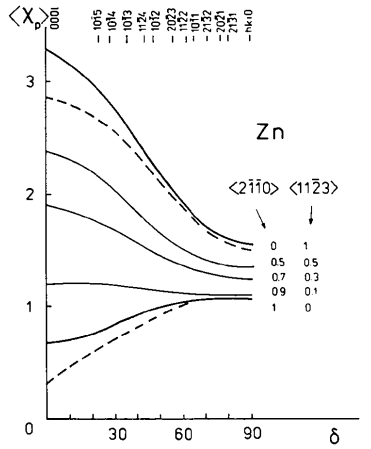

Fig. 3. Dependence of average orientation factors $\langle \chi_p \rangle$ on the angle δ for various fractions of dislocations with the Burgers vectors $a/3$ ⟨2$\bar{1}\bar{1}$0⟩ and $a/3$ ⟨11$\bar{2}$3⟩ for zinc. The dashed lines correspond to the approximation of elastic isotropy for the limiting cases (fractions 0, 1).

calculated by *HEXAN* and those obtained in the approximation of isotropy have to be taken into account for dislocations with Burgers vectors $a/3$ ⟨2$\bar{1}\bar{1}$0⟩ on basal planes, but especially for dislocations of the slip systems ⟨11$\bar{2}$3⟩ {11$\bar{2}$2}. For the latter the dependence of the factor χ is completely different in the cases of elastic isotropy and anisotropy even in materials with a low degree of elastic anisotropy. Accordingly the reflections 000*l* and *hki*0 are strongly affected by the elastic anisotropy.

(iii) According to Figs. 1 and 2 (compare also the upper and the lower curves of Fig. 3) the anisotropy of the X-ray diffraction line broadening can be relatively high if the scattering crystal contains dislocations with special Burgers vectors. Nevertheless, for dislocation arrangements with several types of Burgers vectors, as, for instance, $a/3$ ⟨2$\bar{1}\bar{1}$0⟩ and $a/3$ ⟨11$\bar{2}$3⟩, the effect rapidly decreases. This is illustrated by the intermediate curves of Fig. 3 calculated for various fractions of the Burgers vectors just mentioned. As follows from (9) the influence of the Burgers vector $a/3$ ⟨11$\bar{2}$3⟩ is more pronounced, in this connection, because of its greater magnitude.

From Figs. 1, 2 and 3 it can immediately be understood that reflections of the type *hki*0 or *hkil* with $h, k \gtrsim l$ (*e.g.* 10$\bar{1}$0, 11$\bar{2}$0, 20$\bar{2}$0, 21$\bar{3}$0, 20$\bar{2}$1, 21$\bar{3}$1) have nearly equal orientation factors and are very insensitive to special features (kind and fraction of Burgers vectors, dislocation character) and, moreover, to the elastic anisotropy of the scattering crystal. Such diffraction peaks are consequently suitable for the estimation of dislocation densities in particular. Furthermore they can be used for the separation of different components of the physical line broadening instead of different reflection orders, the measurement of which is usually difficult in non-cubic materials. On the other hand, reflections of the kind 000*l* or *hkil* with $h, k \ll l$ (*e.g.* 0002, 1014, 1015, 0004, 1124) have significantly different orientation factors and depend sensitively on the relative content of dislocations with different Burgers vectors. Therefore, if we take into account the effect of elastic anisotropy as described above, the line broadening of such reflections can be used for an estimation of the fractions of dislocations with different Burgers vectors (*e.g.* $a/3$ ⟨2$\bar{1}$0⟩ and $a/3$ ⟨11$\bar{2}$3⟩) being present in the scattering material. Strictly speaking the effect of X-ray line-broadening anisotropy can be used for this purpose. Since dislocations with Burgers vectors $a/3$ ⟨2$\bar{1}$0⟩ are favoured in most hexagonal materials (*e.g.* Predvoditelev & Troickyi, 1973) the broadening of the 000*l* reflections should be smaller than that of the other diffraction peaks. In the literature no unambiguous indication of the effect could be found, though. This is probably because dislocations with Burgers vectors $a/3$ ⟨11$\bar{2}$3⟩ have a much greater influence on the X-ray diffraction profiles and a relatively small fraction of them is sufficient to cause a significant line broadening. Hence, in crystals with a high degree of elastic anisotropy like zinc or cadmium, for example, an increasing content of such dislocations would lead to a line broadening of the 000*l* reflections much higher than that of the other diffraction peaks (Fig. 3).

In order to check this, investigations on plastically deformed zirconium were performed. The results will be presented in a final paper III.

References

Chung, D. H. & Buessem, W. R. (1968). *Anisotropy in Single Crystal Refractory Compounds*, edited by F. W. Vahldiek & S. A. Mersol, pp. 217–245. New York: Plenum.

Klimanek, P. & Kužel, R. Jr (1988). *J. Appl. Cryst.* **21**, 59–66.

Krivoglaz, M. A. (1967). *Teoriya Rasseyaniya Rentgenovskich Luchey i Neytronov Realnymi Kristallami.* Moscow: Nauka. Engl. transl. *Theory of X-ray and Thermal Neutron Scattering by Real Crystals* (1969). New York: Plenum.

Krivoglaz, M. A. (1983). *Difrakciya Rentgenovskich Luchey i Neytronov v Neidealnych Kristallach.* Kiev: Naukova Dumka. Engl. transl. in preparation.

Landolt-Börnstein (1966). *Numerical Data and Functional Relationships in Science and Technology.* Group III, Vol. 1. Berlin: Springer-Verlag.

Lee, M. & Gilmore, R. S. (1982). *J. Mater. Sci.* **17**, 2657–2660.

Predvoditelev, A. A. & Troickyi, D. A. (1973). *Dislokacii i Tochechnyie Defekty v Gegsagonalnych Metallach.* Moscow: Atomizdat.

Raychenko, A. T. & Martynova, I. F. (1970). *Metallofizika*, **31**, 126–132.

Steeds, J. W. (1973). *Introduction to Anisotropic Elasticity Theory of Dislocations.* Oxford: Clarendon Press.

Teodosiu, C. (1982). *Elastic Models of Crystal Defects.* Editura Academiei Bucuresti. Berlin: Springer-Verlag.

Wilkens, M. (1970). *Phys. Status Solidi A*, **2**, 359–370.