STRUCTURAL INVESTIGATION OF TWINNED CRYSTALS R. A. TAMAZYAN, YU. A. MALINOWSKII, AND V. I. SIMONOV Institute of Crystallography, USSR Acad. Sci., Moscow

INTRODUCTION

The phenomenon of complex crystals of the same substance has been known to mineralogists for a long time. These complex crystal objects consisting of two or more components whose mutual orientation is strictly fixed were called twins. The twins may be distinguished from single crystals by their morphological features. It should be noted that the morphology does not always allow one to distinguish a twin from a single crystal. New methods of studying twins should be applied. Twin domains can differ in their optical, mechanical, electric, magnetic and other physical properties, if anisotropy of the appropriate characteristics is noninvariant relative to twinning symmetry operation.¹ To distinguish a twin from a single crystal by measuring some of its physical properties, one should compare the measured values of tensor elements of the given property with the values of appropriate tensor elements of the same physical property of a single crystal. However, sufficient information on the properties of the single crystal which is being studied is not always available. Then, optical methods are preferable. For instance, if the characteristics of an optical indicatrix are not invariant relative to symmetry operation of twinning, the twin domains differ from each other in polarized light. If the dimensions of the twin domains are microscopically small (such twins are called microtwins), they will be indistinguishable at the given spatial resolution.

In case of microtwinning, to distinguish twins from single crystals one can make use of X-ray diffraction methods, because in this case domain sizes are not important, it is only the total volume of the domains that matters. As is known, all crystals have a mosaic structure and consist of blocks measuring $10^{-4} - 10^{-5}$ cm, that scatter X-rays incoherently.² It is just owing to such crystal structure that kinematic theory of X-ray diffraction can be applied to X-ray structural analysis. In case of twins with random boundaries the scattering of X-rays by various componets will be incoherent. Therefore, a diffraction pattern from a twined crystals is a superposition of diffraction patterns from their components. It may seem that diffraction methods are ideal for investigating twins, because different twin domains could be readily distinguished on the diffraction pattern, and, thus, one could distinghish twin from a single crystal. However, very often a symmetry element of twinning is a symmetry element of the lattice (it transforms the lattice into itself or in a parallel position) or a sublattice of a twin domain. In such cases the diffraction pattern of twin does not differ geometrically from a diffraction pattern of single crystal. There is difference only in reflection intensities. Diffraction patterns of such twins are often erroneously interpreted as diffraction patterns of single crystals. As a result, either the analysis of the atomic structure is impossible or the author obtains a wrong results of the X-ray structural study: the space group and atomic model of the crystal are determined incorrectly. The results reported in some structural papers are doubtful from the point of view of not taking into account microtwinning. In this connection, we would like to stress how important is to determine reliably and in time the microtwinning in the samples studied.

Symmetry and classification of twins

Since the mutual orientation of twin domains obeys certain laws, it is convenient to make use of the group theory. Black-and-white Shubnikov symmetry groups may be used to describe the twins.³ While characterizing a twin using the black-and-white groups, it is natural to relate the symmetry elements connecting different twin domains with symmetry elements of the group, that change their colour. Symmetry elements of the initial single crystal should then be related to usual symmetry elements. The problems of symmetry of twins were discussed in detail in a monograph by V. A. Mokievskii "Morphology of Crystals".⁴ In that paper the following determination of a twin is given: "A twin is such a conglomerate ot two similar components whose symmetry group is a supergroup of index 2 of subgroups of components symmetry class". According to this determination the symmetry of twing G' may be obtained by direct or semidirect product of twinning symmetry element g' and symmetry class of twin component K:

$$G' = g' \otimes K.$$

Therefore, the following relationships are true:

$$G' \supset H \subseteq K$$

$$N_{C'} = 2 \cdot N_{U'}$$
(1)

where H is a subgroup of crystal class K, which is invariant with respect to twin symmetry element g', $N_{G'}$ and N_{H} are orders of groups G' and H, respectively. It should be noted that in a particular case, when G' is a direct product of g' and K, H is a trivial subgroup of K (H = K). According to the above determination of twins the authors of paper⁴ obtained all 58 symmetry groups for the crystallographic twins.

Proceeding from twin symmetry V. A. Mokievskii suggests to split twins into two groups: 1) rigid twins (twins where symmetry element is rigidly related with the crystal) and 2) non-rigid twins (twins in which the orientation of twinning symmetry element is ambiguously determined). For instance, a twin with the symmetry 4/m' of a crystal of class 4 is rigid, because the orientation of (001) twining plane is unambiguously determined. A twin with 4m'm' symmetry of a crystal of the same class is nonrigid, because twinning over any of (hk0) planes leads to the same twin symmetry.

The derivation of all symmetry groups and classification of twins on the basis of symmetry do not take into account the orientation of a twinning symmetry elements with respecting to componnent lattice. However, in the course of X-ray structural studies the orientation of twinning symmetry elements in a crystal lattice is of major importance, because it is responsible for the influence of twinning on the diffraction pattern.

Supposing, twin components have lattices L' and L" which can be described by primitive unit cells built on triplets of noncomplanar vectors a'_i and a''_i , respectively. Let the translation vectors of lattice L" be connected with translation vectors of lattice L' by a linear combination:

$$a_i^{"} = \sum_j t_{ij} a_j^{'}$$
(2)

where t_{ij} are elements of matrix T, characterizing twinning symmetry. Depending on the twinning symmetry element and its orientation respecting to component lattice, L' and L" lattices can have a common nodal raw, common nodal plane and common sublattice Γ . In particular case, Γ can coincide with L' and L" lattices. According to these three possibilities, we distinguished "one-dimensional periodic", "two-dimensional periodic" and "three-dimensional periodic" twins respectively.^{5,6} In "three-dimensional periodic" twins there exists a lattice Γ , in which translation vectors

are the translation vectors for all the twin domains. This lattice Γ should, naturally, be considered as the latice of the twin and it has been called twin lattice. There are no particular problems in the methods of X-ray structural analysis of "one-dimensional periodic" or "two-dimensional periodic" twins. The diffraction pattern from such twins contains split reflections that readily indicate twinning, and it is possibile to measure diffraction reflections from the separate twin domains quite easily. Therefore below only "three-dimensional" periodic twins will be considered.

It was G. Friedel who was the first one to tackle the problem of the interrelation between twinning symmetry elements and the crystal lattice of the initial single crystal.⁷ Proceeding from the fact that the components of "three-dimensional periodic" twins have coinciding or almost coinciding sublattices, he formulated four alternative conditions one of which the translation latice of a twin domain should satisfy:

1) the translation lattice possesses symmetry elements which are absent in the structural motif;

2) the translation lattice possesses elements of pseudosymmetry;

3) a simple lattice multiple to the translation one is characterized by symmetry elements that are absent in the translation lattice itself;

4) a simple lattice multiple to the translation one is characterized by pseudosymmetry elements.

Depending on a particular condition realized, G. Friedel singles out four types of twinning: 1) twinning by merohedry, 2) twinning by pseudomerohedry, 3) twinning by reticular merohedry and 4) twinning by reticular pseudomerohedry.

The centre of inversion, crystallographic axes and a mirror symmetry plane are the elements of twinning symmetry. Twinning by the inversion centre almost leads to twinning by merohedry, because any translation lattice obeys the inversion centre. Twinning by crystallographic axes or mirror symmetry planes, depending on their orientation in the lattice, can lead to any of the four types of twinning.

In order to have L' and L" lattices with coinciding or almost coinciding Γ ' and Γ " sublattices, the twinning symmetry element should be a symmetry or pseudosymmetry element of Γ ' and Γ " sublattices. Consequently, twinning symmetry axes should coincide with the nodal raw perpendicular or almost perpendicular to the nodal plane of L' and L" lattices. In the same way, twinning symmetry planes should coincide with the nodal raw of L' and L" lattices. In case the condition of the perpendicularity is roughly fulfilled, twinning by pseudomerohedry or reticular pseudomerohedry will take place. G. Friedel suggested to introduce the parameter ω , characterizing twins by pseudomerohedry. Twin aperture ω is the angle between

the nodal raw and the normal to the nodal plane. However, we readily see that twinning by pseudomerohedry with aperture $\omega = 0$ is possible. Let us consider, for example, orthorhombic single crystals with the ratio $a:b\approx 1$, twinned by a four-fold axis coinciding with the c axis of the single crystal. In this case the twinning axis is strictly perpendicular to the nodal plane (001), consequently, $\omega = 0$, nevertheless this is twinning by pseudomerohedry, as L' and L" coincide only roughly.

Thus, the condition of the nodal raw being perpendicular to the nodal plane is not a strict criterion for distinguishing between twinning by merohedry and twinning by pseudomerohedry, it is correct only in the case when twinning symmetry elements are second-order elements. A strict criterion for determining twinning by merohedry or by pseudomerohedry was offered by A Santoro.⁸ He suggested the matrix Δ_{ij} as a criterion of pseudomerohedry or reticular pseudomerohedry of the twinning law. This matrix is determined in the following way:

$$\Delta_{ij} = \left(\sum_{m, n} t_{im} t_{jn} A_{mn} - A_{ij}\right) / \sqrt{A_{ii} A_{jj'}}$$
(3)

where t_{ij} are elements of the twinning symmetry matrix, A_{ij} are elements of the metrical tensor of the lattices L' and L" and they are determined in the following way:

$$A_{ij} = a_i \cdot a_j.$$

If all the components of Δ_{ij} are equal to zero, twinning by merohedry takes place. If at least one of them differs from zero, twinning by pseudomerohedry will take place. The Δ_{ij} matrix is an analogue of twin aperture and demonstrates the extent of the coincidence of L' and L" lattices.

In case Γ' and Γ'' lattice fully coincide, the lattice $\Gamma = \Gamma' = \Gamma''$ is, undoubtedly, a twin lattice. In the case of twins by pseudomerohedry none of the Γ' or Γ'' lattices, strictly speaking, are translational for both components simultaneously. However, in this case the translation lattice which is in the best agreement with both Γ' and Γ'' lattices can be regarded as a twin lattice. Apparently, the non-complanar vectors of that lattice characterizing the primitive unit cell are determined in the following way:

$$r_i = (r'_i + r''_i)/2,$$
 (4)

where r'_i and r''_i are basic vectors of Γ ' and Γ " latices respectively. We see from (4) that such a determination of a twin lattice is good both for twins by merohedry (it coincides with Γ ' and Γ " lattices) and for twins by pseudomerohedry (it is better than any other lattice discribing translations of all twin domains). It should be noted that in the course of X-ray structural analysis, a twin lattice acquires a peculiar sense, because the experimental data obtained from a twin are indexed just according to this lattice.

The volume ratio of a primitive cell of the twin latitce Γ to the volume of a primitive cell of single crystal lattice L is called a twin index.⁹

$$\mathbf{n} = \mathbf{V}_{\Gamma} / \mathbf{V}_{\mathrm{L}} \tag{5}$$

The twin index is an important parameter that characterizes a diffraction pattern from a twin. We see from the determination of the twin index (5) that this parameter allows one to distinguish between twinning by merohedry and twinning by reticular merohedry. Proceeding from this, G. Donnay suggested a new classification of twins: twinning by TLS (twin lattice symmetry) – where a symmetry element of twinning is a symmetry element of the lattice or sublattice of a twin domain and TLQS (twin lattice quazi-symmetry), that is twinning by a pseudosymmetry element of the lattice or sublattice of a twin domain.¹⁰ We readily see that twinning by TLS includes twinning by merohedry (twin index equal to unity) and twinning by reticular merohedry (twin index exceeds unity), while twinning by TLQS combines twinning by pseudomerohedry (twin index si equal to unity) and twinning by reticular pseudomerohedry (twin index exceeds unity).

Thus, all the above parameters characterizing twins are of principal importance during X-ray structural studies, because they determine peculiar features of diffraction patterns from twins.

X-RAY STRUCTURAL STUDIES OF TWINS

X-rays are diffracted by twin domains incoherently, therefore, the diffraction pattern from a twin is a superposition of diffraction patterns from twin domains related by twinning symmetry element. Since "three-dimensional periodic" twins have a twin lattice, whose translation vectors are translation vectors for the all domains, then their diffraction patterns may be indexed according to this twin lattice. Thus, the intensity of a beam diffracted from a unit volume of a twin I(H), will have the following form in each node of the reciprocal twin lattice H:

$$I(H) = \alpha \cdot J_1(H) + (1 - \alpha) \cdot J_2(H), \tag{6}$$

where α is volume relation of the first twin domain to the twin volume, $J_1(H)$ and $J_2(H)$ are intensities of X-rays diffracted from unit volumes of

the first and second domains, respectively. During the data collection the total intensities I(H) are collected, whereas to make the structural analysis and carry out correct X-ray structural studies it is necessary to separate the contributions of twin domaines J(H) in the total intensity. In order to separate contributions made by twin domains additional information, besides equation (6) is required.

First of all, the question about symmetry of the diffraction pattern of the twin should be answered. Since the diffraction pattern of the twin is a superposition of diffraction patterns of single-crystalline twin domains, it also obeys the Friedel law (without account of anomalous scattering). Consequently, the symmetry of the diffraction pattern of twins without account of anomalous scattering is characterized by those symmetry groups of twins which contain a centre of inversion. There are 10 such groups. In cases when twin symmetry is within one of these ten groups the symmetry group of the diffraction pattern coincides with that of the twin. In order to determine symmetry group of diffraction pattern of the twins whose symmetry groups do not contain a centre of inversion, such a centre should be added to the symmetry group. However, 21 out of 58 twocoloured symmetry groups of twins contain a black-and-white inversion centre that is responsible for twinning. An addition of a usual centre of inversion to these groups makes them single-coloured and leads to 11 centrosymmetric single-coloured groups that coincide with the Laue classes. These groups describe those twins in which a twinning symmetry element is a Laue class symmetry element. They are listed In Table 1. If diffraction

twin symmery	Symetry of diffraction pattern of a twin	
ī,	ī	
2'/m, 2/m'	2/m	
m'm'm', mmm'	mmm	
3'	3	
$\overline{3}$ 'm, $\overline{3}$ 'm'	$\overline{3}m$	
4'/m', 4/m'	4/m	
4/m'mm, 4/m'm'm', 4'/m'mm'	4/mmm	
6/m', 6'/m	6/m	
6/m'mm, 6/m'm'm', 6'/mmm'	6/mmm	
m' 3 '	m 3	
$m'\overline{3}'m, m'\overline{3}'m'$	m3m	

Table 1. Symmetry groups of twins with Laue-class symmetry element as twinning symmetry element.

patterns of these twin domains are superimposed equivalent reflections overlap. Substituting $J_1(H) = J_2(H)$ into (6), we obtain:

$$I(H) = J(H) \tag{7}$$

We see from (7) that the diffraction patterns from such twins do not differ from diffraction patterns of the separate twin domaines. Hence, it is impossible to determine such twins by X-ray diffraction analysis. It should be noted that, since a twinning symmetry element is a symmetry element of the Laue class for these twins, it is also a symmetry element of the lattice of the twin domain. Hence, according to G. Friedel classification they are twins by merohedry. The authors of¹¹ refer them to twins by merohedry of class I. In the same paper all the symmetry classes which can from twins by merohedry of class I are listed and the influence of such twinning on the diffracton pattern is analyzed.

However, during the analysis of a diffraction pattern, only the Laue class (the symmetry relating reflections with the same intensity) are determined, but not the symmetry group of the diffraction pattern of the twin. It follows from (6) that the Laue class of the diffraction pattern depends on twin volume ratio. For the same symmetry group of diffraction patterns, depending on the value of the parameter α ($\alpha = 0.5$ or $\alpha \neq 0.5$). Table 2 presents 37 symmetry groups of twins that can be distinguished by X-ray diffraction patterns of twins and, their Laue classes depending on the value of diffraction different groups of diffraction analysis. They are united according to symmetry groups of diffraction patterns of twins and, their Laue classes depending on the value of α are indicated for each group. To solve the equation (6) one should de-

Twin symmetry	Symmetry of the diffraction pattern	Laue class at $\alpha = 0.5$	Laue class at $\alpha \neq 0.5$
2', m', 2'/m'	2'/m'	2/m	$\overline{1}$
m'm'2, m'2'm, 2'2'2, m'm'm	m'm'm	mmm	2/m
3m', 32', <u>3</u> m'	3m'	3m	3
4', 4 ', 4'/m	4'/m	4/m	2/m
4'22', 4'm2', 4'2m', 4'mm', 4'/mmm'	4'/mmm'	4/mmm	mmm
4m'm', 42'm', 42'2', 4/mm'm'	4/mm'm'	4/mmm	4/m
6', 6 ', 6'/m	6'/m'	6/m	3
6'2m', 6'm2', 6'mm', 6'22', 6'/m'mm'	6'/m'mm'	6/mmm	$\overline{3}m$
62'm', 6m'm', 62'2', 6/mm'm'	6/mm'm'	6/mmm	6/m
4'3m', 4'32', m3m'	m 3 m'	m3m	$m\overline{3}$

Table 2. Correlation between twin symmetry and the appropriate diffraction symmetry

termine just the symmetry group of diffraction pattern of a twin, but not the Laue class. We see from Table 2 that no more than two symmetry groups of a diffraction pattern of a twin correspond to each Laue class. Taking into account the fact that twin volume ratio is a ramdom factor (depending on growth conditions, thermal treatment etc.), after we obtain diffraction data from several different samples, we can assume with a great degree of probability that twin volume ratio for at least one of them will differ from 0.5. Thus, ambiguous determination of the symmetry of the diffraction pattern of a twin is retained only for the Laue classes $\overline{3}$ and 2/m. However, as mentioned in the previous section, a twinning symmetry element is a symmetry element of a twin lattice. Consequently, in cases of ambiguously determined symmetry of the diffraction pattern of a twin it should be analyzed with account of the symmetry of a twin lattice. For instance, if the Laue class of the diffraction pattern is 2/m and $\alpha \neq 0.5$, the symmetry of the diffraction pattern from the twin is either 4'/m or m'm'm (see table 2). If the symmetry of a twin lattice is mmm, the symmetry group of the diffraction pattern of a twin is unambiguously determined as m'm'm, for the symmetry of twin lattice 4/mmm the symmetry of the diffraction pattern of a twin can be described both by symmetry group 4'/m and m'm'm. If the Laue class of the diffraction pattern is 3, the symmetry of the diffraction pattern of a twin is either 3m' or 6'/m' (see table 2). Similarly to the above example, in this case, if the symmetry of the twin lattice is 3m, the symmetry group of the diffraction pattern of a twin is determined unambiguously, while for symmetry of a twin lattice 6/ mmm there is ambiguity, and both possible symmetry groups of the diffraction pattern of a twin should be analyzed. The use of the data listed in Table 1 and 2 facilitates the determination of symmetry group of the diffraction pattern of a twin.

Supposing, the symmetry of a diffraction pattern of a twin is described by the symmetry group G', and the reciprocal twin lattice vectors H_1 and H_2 are related by a twinning symmetry element. Then the intensities of diffracted beams I_1 and I_2 in nodes H_1 and H_2 , respectively, are as follows:

$$I_1 = \alpha \cdot J_1 + (1 - \alpha) \cdot J_2$$

$$I_2 = \alpha \cdot J_2 + (1 - \alpha) \cdot J_1.$$
(8)

In the general case, if the twin volume ratio is unknown, the system of equations (8) cannot be solved. To determine twin volume ratio D. Briton suggested the following technique.¹² Proceeding form (8) let us calculate the following relationship:

$$I_1/(I_1 + I_2) = [1 - \alpha \cdot (1 - J_1/J_2)]/(1 + J_1/J_2).$$
(9)

The values J_1 and J_2 are independent and assume only positive values, consequently, the relationship J_1/J_2 assumes values within the interval $[0, \infty]$. The dependence of $I_1/(I_1 + I_2)$ on J_1/J_2 is presented in Fig. 1. We see from Fig. 1 that as the argument changes within $[0, \infty]$ the function takes values within $[\alpha, 1 - \alpha]$. It is impossible to construct the plot of the dependence $I_1/(I_1 + I_2)$ on J_1/J_2 from X-ray diffraction data, but, calculating the relationship $I_1/(I_1 + I_2)$ for all pairs of reflections that are related by a twinning symmetry operation, we can determine the limit values of the function, and, consequently, the twin volume ratio. Then, the system of equations (8) can be solved using a defined value α .

In¹³ P. Murrey-Rust suggests another technique for finding the twinvolume ratio, that slightly differ from the Briton method. The following relation between I_1 and I_2 values can be determined from (8):

$$I_{1} = \{ [\alpha + (1 - \alpha) \cdot J_{2}/J_{1}] / [(1 - \alpha) + \alpha \cdot J_{2}/J_{1}] \} \cdot I_{2}.$$
(10)

It is seen from (10) that I_1 , at a certain value of I_2 , depending on J_2/J_1 , can take values in the range $[\alpha I_2/(1-\alpha), (1-\alpha) I_2/\alpha]$. If we construct an experimental plot of the dependence of I_1 on I_2 , all the points of the plot will

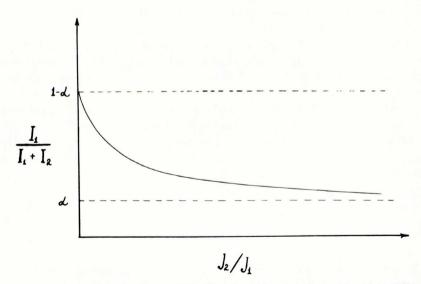


Fig. 1. Dependence of $I_1/(I_1 + I_2)$ on J_1/J_2 , where I_1 and I_2 are the intensities of the diffracted beams in nodes related by twinning symmetry operation, J_1 and J_2 are the contributions of twin domains into the total intensity.

lie inside the sector formed by direct lines $I_1 = [\alpha/(1-\alpha)] \cdot I_2$ and $I_1 = [(1 - \alpha)/\alpha] \cdot I_2$ (Fig. 2). Calculating the angle θ between the direct lines, that determine the sector and the ordinate axis the value of α can be found according to the following formula:

$$\alpha/(1-\alpha) = \operatorname{tg} \varphi \tag{11}$$

Another procedure, that differs from the above one was suggested by P. Fisher abd R. Sweet.¹⁴ They solved the system of equations (8) relative to J_1

$$\mathbf{J}_1 = [\mathbf{I}_2 - \alpha \cdot (\mathbf{I}_1 + \mathbf{I}_2)] / (1 - 2 \cdot \alpha). \tag{12}$$

Substituting a definite value of α within the range [0,0.5] into equation (12) we construct an experimental plot N(α), where N(α) is the number of J₁ that at a given value α takes negative values. If the true value of α is α_0 , the function N(α) within the interval [0, α_0] will take a zero value, while in the interval [α_0 , 0.5] the value of function will increase abruptly (Fig. 3). Using the plot N(α) the value of α_0 is determined and at a given value α_0 the contributions of twin domains J(H) in total intensity I(H) of overlapped reflections are determined.

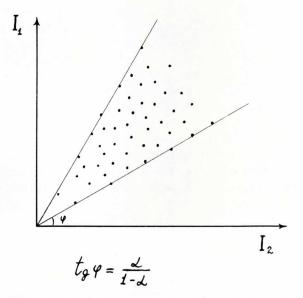


Fig. 2. Experimental dependence I_1 on I_2 , where I_1 and I_2 are the intensities of diffracted beams in nodes, related by twinning symmetry operation.

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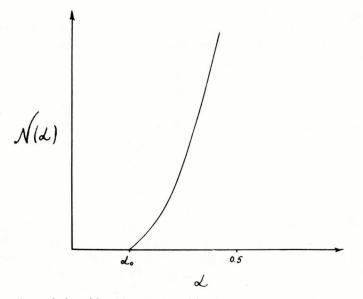


Fig. 3. Experimental plot of funcition $N(\alpha)$ used for determining twin volume ratio.

It is not sufficient to solve the equation (6) for an unambiguous X-ray study. In order to do this we should determine the lattice of the single crystalline twin domaine and the symmetry of its diffraction pattern. If the twin index is n = 1, this problem becomes trivial, because the component lattice coincides with the twin lattice, while the symmetry of the diffraction pattern from a twin. In the case the twin index differs from unity, the twin lattice is only a sublattice of the component lattice. In this case a primitive cell of a twin lattice contains (n - 1) nodes of the component lattice. Therefore, when the diffraction pattern of a twin domaine is described according to a twin lattice then the (n - 1)/n part of nodes of the twin reciprocal lattice is, in fact, zero, as there appear absences due to the centering of the unit cell of twin lattice. When the diffraction patterns of the twin domains are superimposed, some zero nodes of the diffraction pattern of one domaine coincides with the reflections from another domaine, while the other nodes coincide with the same zero nodes of another domaine. This is the reason for the appearnce of peculiar "twin" absences at n = 1that should assist the investigator in finding the component lattice and in determining the symmetry of the diffraction pattern of twin domaine. It should be noted that at n = 1 the symmetry of the diffraction pattern of a twin domain is not always described by a monochromatic subgroup of the symmetry group of the diffraction pattern of a twin. If the absences

due to the space group of the single crystal lie within certain planes or on certain axes, twin absences are arranged in three-dimensional reciprocal space. That is why the analysis of the diffraction pattern of a twin carried out with the aim of determining the component lattice proceeding from twin absences, is a complicated problem. In fact, this problem can be solved effectively by applying the function of interatomic vectors.¹⁵

Abstract

Methods of structural investigations of twinned crystals are described in this paper. The problems of classification of twins based on twin domains symmetry and relations between their lattices are considered. The problem of reciprocal space symmetry of twinned crystals is also considered. The results of such consideration are listed in two tables, these may be usefull for the determination of possible single crystal symmetry from analysis of twinned crystals diffraction pattern.

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