ture being composed of virtually planar molecules arrayed in planes extending throughout the crystal and stabilized by $\pi - \pi$ interactions – a graphitic type of structure perhaps further stabilized by interaction of charge separations associated with the lactone group. This possibility offers an attractive speculation concerning the intermolecular bonding and the role of lactone groups in stabilizing carbonaceous structures (see Garten & Weiss, 1957).

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The Direct Structure Determination of a Silicon Carbide Crystal of Type 120 *R*

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The direct method of determining polytypic structures, recently published independently by Tokonami and by Farkas-Jahnke, has been applied successfully to a SiC crystal of type 120*R*, whose lattice constants are $a = 3\cdot080 \pm 0\cdot003$, $c = 302\cdot4 \pm 0\cdot1$ Å when described on hexagonal axes. The Zhdanov symbol of the structure is $[32(22)_5\ 32\cdot22\cdot33]_3$. It is shown that this direct method applies whenever sufficiently accurate intensity data are available. The required accuracy has been estimated; it increases with the number of double layers in the unit cell.

Introduction

Recently, a direct method to solve the crystal structures of polytypic substances such as SiC and ZnS has been developed independently by Farkas-Jahnke (1966) and Tokonami (1966). In this paper we shall, for the most part, use the notation of the former.

In short, the method is as follows. A set of observed intensities $|F(01l)|^2$ with $l = 1,2 \ldots n$ is converted into a set of $|S(01l)|^2$ values. For the above substances the relevant equations are

$$|F(01l)|^2 = \alpha[f_I^2 + f_{II}^2 + 2f_I f_{II} \cos (3\pi l/2n)]\ |S(01l)|^2,$$

$$\sum_{l=1}^{n} |S(01l)|^2 = N^2,$$

where

$\alpha = $ scaling factor,
$f_I, f_{II} = $ scattering factors of the two atomic species,
$n = $ number of double layers in the hexagonal unit cell,
$N = n$ for trigonal and hexagonal structures,
$\quad = n/3$ for rhombohedral structures .

The data are now used to compute the Patterson-like function

$$\Pi(m,p) = \frac{N}{3} + \frac{2}{3N} \sum_{l=1}^{n} |S(01l)|^2 \cos 2\pi \left(\frac{m}{3} + \frac{pl}{n} \right)$$

at the points $m = 0, \pm 1$; $p = 0,1,2 \ldots N/2$ [or $(N-1)/2$ if N is odd]. It is easily shown that $\Pi(m,p)$ represents

the frequency of occurrence of the stacking vector $[-m/3, m/3, p/n]$ in the structure. Theoretically, *i.e.* when based upon errorless intensity observations and correction factors, the Π-function has the following properties:

(1) $\Pi(m,p)$ is a non-negative integer (m,p integer)
(2) $\Pi(0,1)=0$ (no two consecutive double layers are located on the same threefold axis)
(3) $\Pi(0,2)$ is even ($=$ number of stacking reversals)
(4) $\sum_m \Pi(m,p) = N$
(5) $\Pi(1,p) - \Pi(-1,p) = p$ (mod. 3) for rhombohedral structures in obverse setting;
$\qquad\qquad = 0$ (mod. 3) for hexagonal and trigonal structures.

The validity of (1) is evident, since each stacking vector occurs an integral number of times. (2) and (3) were given by Tokonami, (4) by Farkas-Jahnke. Equation (5) was derived by the author (*Appendix*).

Procedures for deriving the stacking sequence from the computed values of $\Pi(m,p)$ have been given by both of the above authors. However, there is an inherent difference of opinion as to the required accuracy of the experimental data in their approach.

Farkas-Jahnke tacitly assumes that the experimental errors are small. In that case the theoretical and the experimental $\Pi(m,p)$ values are almost identical and her way of interpreting these values leads elegantly and fairly straightforwardly to the correct structure.

Tokonami argues that the order of weak and strong reflexions of any one polytypic structure is highly characteristic of that structure (see: Hosoya & Tokonami, 1967). Semiquantitative intensity data, even uncorrected for the continuous factors (Lorentz, polariza-

tion and atomic scattering factors), would suffice for a structure determination, since they constitute a recognizable transform of the structure. Of course, the experimental $\Pi(m,p)$ values computed from such data do not meet the above conditions (1) to (5), but according to Tokonami there would exist a reliable method of computing the true $\Pi(m,p)$ values, whereby the experimental data are refined while the characteristic intensity distribution is preserved.

The direct determination of a 120R type structure of SiC provides a welcome opportunity for comparing the merits of these two approaches.

Experimental

In a batch of SiC crystals, prepared in this laboratory by Dr W.F.Knippenberg, a colourless, transparent specimen was found with linear dimensions in all directions of about 0·2 mm.

Using the 'PAILRED' diffractometer this crystal was found to consist mainly of an unknown 120R type structure ($a = 3·080 \pm 0·003$; $c = 302·4 \pm 0·1$ Å) in syntactic coalescence with a minor quantity of 27R. In addition some very weak difffraction maxima were observed pointing to the presence of small amounts of other SiC structures which could not, however, be identified. There were no indications of one-dimensional disorder. The reflexions of 120R were easily indexed by comparing their positions in the diagram with those of 27R. At the same time it was verified that the presence of the latter structure would not interfere with the structure determination of the former. No certainty could be obtained about the influence of the unidentified structures; some of their reflexions might coincide with those of 120R, but, to judge from

Table 1. *The experimental values of $\Pi(m,p)$ and those computed for the structures (a), (b) and (c)*

	$\Pi_{exp}(m,p)$			$\Pi_a(m,p)$			$\Pi_b(m,p)$			$\Pi_c(m,p)$		
m	-1	0	1	-1	0	1	-1	0	1	-1	0	1
p												
0	0·0	40·0	0·0	0	40	0	0	40	0	0	40	0
1	19·9	1·3	18·8	21	0	19	21	0	19	21	0	19
2	10·8	16·4	12·8	10	18	12	10	18	12	10	18	12
3	17·4	5·3	17·3	18	4	18	18	4	18	18	4	18
4	2·6	31·3	6·2	2	32	6	2	32	6	2	32	6
5	24·0	1·1	14·9	25	0	15	25	0	15	25	0	15
6	7·1	20·5	12·4	7	20	13	7	20	13	6	22	12
7	17·8	5·6	16·5	18	6	16	18	6	16	19	4	17
8	3·1	26·6	10·3	4	27	9	4	27	9	2	28	10
9	26·4	1·8	11·8	25	2	13	25	2	13	27	1	12
10	4·9	21·5	13·7	4	22	14	4	22	14	4	22	14
11	19·5	6·4	14·1	19	6	15	21	5	14	21	5	14
12	4·2	23·7	12·1	5	24	11	3	25	12	3	25	12
13	24·2	4·7	11·1	23	5	12	25	4	11	25	4	11
14	4·0	19·5	16·5	3	20	17	3	20	17	3	20	17
15	23·5	6·0	10·4	23	6	11	25	5	10	25	5	10
16	3·4	21·9	14·7	4	22	14	2	23	15	2	23	15
17	24·1	6·6	9·3	23	7	10	25	6	9	25	6	9
18	3·8	18·4	17·7	3	19	18	3	19	18	3	19	18
19	24·2	7·4	8·4	23	8	9	25	7	8	25	7	8
20	3·3	18·4	18·4	4	18	18	2	19	19	2	19	19

the weakness of their observable reflexions, this could give rise only to minor errors.

In order to avoid appreciable absorption errors Mo $K\alpha$ radiation was used for the intensity measurements ($\mu = 15$ cm^{-1}; $\mu d \sim 0.3$). As a consequence the resolution of the diffraction maxima was too poor for integrated intensity measurements to be possible. Therefore, the peak heights were taken as a measure of the integrated intensities, a procedure that seems justified in view of the narrow range of θ-angles ($8°-11°$) of the reflexions involved.

The experimental data were corrected for non-linearity in the counting circuitry and for the Lorentz factor of a stationary crystal, but the absorption correction and the polarization factor were neglected. Using the atomic scattering factors of *International Tables for X-ray Crystallography* (1962), the intensities were converted into $|S(01l)|^2$ values which were subsequently employed to compute $\Pi(m,p)$ (Table 1).

Structure determination

Clearly, the experimental $\Pi(m,p)$ values do not meet the five conditions set out in the *Introduction*. A first impression of the effect of experimental errors is obtained from the fact that $\Pi(0,1) = 1.3$ instead of zero. However, the exact value of $\Pi(0,1)$ is the only one known in advance. In order to estimate the possible errors of the other $\Pi(m,p)$ we now arbitrarily assume that the intensity data have an average error of about 10%. With $\sum\limits_{l=1}^{n} |\delta|S(01l)|^2| / \sum\limits_{l=1}^{n} |S(01l)|^2 = 0.1$ it follows from the definition of $\Pi(m,p)$ that

$$|\delta\Pi(m,p)| = \left| \frac{2}{3N} \sum_{l=1}^{n} \delta|S(01l)|^2 \cos 2\pi \left(\frac{m}{3} + \frac{lp}{n}\right)\right|$$

$$\leq \frac{2}{3N} \sum_{l=1}^{n} |\delta|S(01l)|^2| = 2.67 .$$

Table 2. *The value of* $|S(01l)|^2$.

(o) observed; (e) the same corrected for extinction; (a), (b), (c) computed for the corresponding structures.

l	$|S_o(01l)|^2$	$|S_e(01l)|^2$	$|S_a(01l)|^2$	$|S_b(01l)|^2$	$|S_c(01l)|^2$
2	38.5	33.5	21.4	33.9	34.8
5	1.7	1.5	3.0	0.2	1.5
8	4.6	4.0	7.7	4.8	3.4
11	2.7	2.4	4.3	5.4	2.3
14	2.2	1.9	1.5	2.7	2.0
17	3.5	3.1	2.4	2.0	3.2
20	3.4	3.0	3.0	3.0	3.0
23	5.6	4.8	5.1	4.5	4.7
26	10.6	9.2	9.4	5.7	10.2
29	123.8	125.2	109.2	122.1	126.6
32	141.7	144.2	155.5	140.2	135.0
35	40.2	35.0	45.8	50.3	37.0
38	11.8	10.3	8.8	15.2	11.0
41	15.9	13.8	5.2	1.7	16.7
44	18.3	16.0	6.9	0.8	18.8
47	6.4	5.6	3.8	7.7	4.8
50	4.1	3.6	17.2	25.4	3.0
53	11.0	9.6	27.5	21.0	6.8
56	14.5	12.6	23.4	4.6	15.9
59	652.2	736.6	657.9	701.2	722.5
62	32.5	28.3	67.4	30.1	35.2
65	10.7	9.3	12.3	21.1	7.7
68	3.7	3.2	5.5	15.4	3.5
71	13.0	11.3	10.7	6.3	7.7
74	20.6	17.9	22.3	15.8	22.5
77	9.5	8.3	5.0	8.5	10.5
80	17.0	14.8	13.0	19.0	19.0
83	71.5	62.2	68.9	65.1	68.1
86	72.4	63.0	61.5	52.4	53.9
89	70.1	61.0	58.1	74.0	67.7
92	40.5	35.2	57.3	47.5	39.2
95	23.2	20.2	11.0	12.5	13.8
98	39.6	34.5	23.6	24.3	34.6
101	11.1	9.6	5.7	5.8	12.0
104	2.5	2.1	5.5	6.4	1.7
107	8.4	7.3	15.7	14.8	7.0
110	7.4	6.5	6.8	4.6	3.0
113	5.5	4.8	0.1	2.0	5.4
116	12.7	11.0	2.3	8.4	10.4
119	15.5	13.5	28.4	14.1	14.1

This last value is the maximum error, which is very unlikely ever to occur. It seems reasonable therefore, to take a possible error of 2 units in each $\Pi(m,p)$ into account.

In practice, as shown already by Tokonami, such uncertainties do not necessarily lead to great difficulties in the structure determination. With $p = 1$, for example, the experimental values are: $\Pi(-1,1) = 19.9 \pm 2$; $\Pi(0,1) = 1.3 \pm 2$; $\Pi(1,1) = 18.8 \pm 2$, but there is only one set of values which meets the required conditions, viz. 21, 0, 19. With $p = 2$ there are two such sets: 11, 16, 13 and 10, 18, 12. On further investigation the first of these turns out to be incompatible with the experimental $\Pi(m,3)$. Thus, even when experimental errors are taken into account, the number of possible values of each $\Pi(m,p)$ is very limited.

Finally, considering all possibilities and following the more systematic Farkas-Jahnke method, three very similar structures are found to be in equally good agreement with the experimental $\Pi(m,p)$ (Table 1). Their Zhdanov symbols are:

(a) $[32(22)_5\ 32\cdot32\cdot23]_3$, (b) $[32(22)_5\ 32\cdot33\cdot22]_3$ and

(c) $[32(22)_5\ 32\cdot22\cdot33]_3$.

When the $|S(01l)|$ for the structures are computed, it is found that (c) is the correct structure; it corresponds to an R index of 6.6% as against 18.1% and 18.0% for (a) and (b) respectively.

Even for this structure there are a few discrepancies between the observed and calculated values of $|S(01l)|^2$ as will be seen in Table 2. Some of the reflexions involved have been remeasured several times under various experimental conditions, but errors in the original measurements were not detected. Since, on the basis of some additional experiments, absorption as well as the Renninger effect were ruled out as the responsible factors, we are inclined to ascribe these discrepancies to the structural impurity of the crystal. In view of the general agreement with experiment and of the great increase of the R index in going from one structure to another closely resembling it, it is considered extremely improbable that the proposed structure would be in error. In this connexion it may be added that similar discrepancies have been found in several other investigations on SiC structures: 105R (Singh & Verma, 1964), 90R (Krishna & Verma, 1963), 24R (Gomes de Mesquita, 1965). Even so, in all these cases there can be little doubt about the correctness of the structure assignment.

Discussion

The fact that the correct structure has been found, out of more than 10^9 possible stacking sequences, demonstrates the power of the direct method of structure determination. The question now is whether or not this method can be applied to elucidate any arbitrary polytypic structure. To answer this question one must

consider that the method works best when all $\Pi(m,p)$ values are unambiguously determined. This would be the case if each $|\delta\Pi| \leq 1$, which means that the average error of the observed intensities should be at most of the order of $3/2N \times 100\%$. In other words, the permissible experimental errors are inversely proportional to the number of double layers in the unit cell. The larger that number, the smaller the chance of a successful analysis.

Of course it might be possible to solve a structure even when there are ambiguities among the $\Pi(m,p)$ values and the present work is an example of this, but then a good deal of luck is needed. This was realized when we tried to redetermine the structure starting from the theoretical $\Pi(m,p)$ for structure (c) and putting $\delta\Pi = \pm 2$. The number of structures to be considered was so large that we abandoned the idea of continuing the analysis to the end.*

Tokonami's method deals exactly with this difficulty. Somewhat simplified, it consists in determining the allowed values of $\Pi(m,1)$ (in the present case: 21, 0, 19) and refining the observed intensities so as to equalize the experimental and calculated $\Pi(m,1)$. Thereby the characteristic intensity distribution is maintained as far as possible. A new table of Π values is computed on the basis of the refined data, from which $\Pi(m,2)$ is now determined. Then the intensity data are refined again and so on. Each time a better starting point is obtained for the continuation of the analysis. In this way it would be possible to determine the true Π values and to solve the structure even when only inaccurate, semi-quantitative data are available. It stands to reason that this procedure requires the intensity distribution of the structure under investigation to be so characteristic that there is no risk of refining the observations until they correspond to another structure. It has been argued by Hosoya & Tokonami that this requirement is generally fulfilled and the method has been applied by Tokonami to elucidate the structure of SiC type 96R. However, a comparison of the observed and calculated intensities (Table 3) shows that their mutual agreement is no better than that of the three sets of calculated intensities of the 120R type structures (a), (b) and (c). A great similarity between the calculated intensity distributions has also been found by Krishna & Verma for two possible structures of SiC type 90R. It seems, therefore, that the availability of accurate intensity data is more essential than hitherto sometimes

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* With $\delta\Pi = \pm 1$ only structure (c) is in agreement with the theoretical Π-function values. With $\delta\Pi = \pm 2$ we find the structures (a), (b) and (c), and in addition we have to consider numerous structures which share the condition that the figure '1' appears in the Zhdanov symbol. Generally, such SiC structures are rejected in advance, as they are assumed to be nonexistent, so that – following common practice – we end up again with structure (c) as the only solution. It would be unsatisfactory, however, to have a method that is inapplicable to the complete set of theoretically possible stacking sequences.

Table 3. *Observed and calculated intensities of* SiC *type* 96R

The observed intensities are those measured directly from a photograph and brought to scale; I_o (10·70) is unreliable. The calculated intensities are actually $|S(10l)|^2$ values. (After Tokonami).

l	$I_o(10l)$	$I_c(10l)$
1	0	0
4	0	0
7	16	2
10	32	5
13	16	11
16	64	27
19	32	21
22	128	23
25	32	19
28	16	8
31	96	60
34	64	126
37	64	36
40	8	11
43	8	0
46	64	31
49	64	256
52	0	12
55	0	1
58	0	0
61	0	1
64	64	289
67	8	1
70	(64)	0
73	8	0
76	32	4
79	96	72
82	16	6
85	8	0
88	16	1
91	8	1
94	0	1

assumed, and that the refinement of observations is rather hazardous.

Even so, a limited use of the above method can certainly be made. In the present study, for example, we could have taken the experimental value $\Pi(0,1) = 1\cdot3$ as an indication and measure of secondary extinction (the only important physical effect for which no corrections were made), and we could have used this value to compute the extinction correction, thereby reducing $\Pi(0,1)$ to zero. When this is done a new set of $|S(01l)|^2$ is obtained (Table 2), to which a new table of $\Pi(m,p)$ values corresponds, none of which deviates by more than one unit from the theoretical value for structure (c). Conversely, with $\delta\Pi = \pm 1$ one finds structure (c) as the only possible solution of the corrected Π-function. Thus the correctness of the structure as well as the extinction correction are confirmed simultaneously. As a result of this correction the R index decreases to 5·9%.

It should be emphasized that this application of Tokonami's method differs from the original one in that a correction is made here for a *known* physical effect and that the magnitude of this correction is determined by the experimental $\Pi(0,1)$, the only $\Pi(m,p)$ whose exact value follows from the theory.

Conclusion

The direct method of determining polytypic structures, especially in the way proposed by Farkas-Jahnke, has been found of great value. It can be applied successfully when sufficiently accurate intensity data are available. The required accuracy increases with the number of double layers in the unit cell, so that the method may be less suitable for structures of the greatest complexity.

Actually, the method consists in a systematic unravelling of the complete set of stacking vectors represented by the function $\Pi(m,p)$. The value of this function can be computed at the relevant points (m,p) on the basis of the experimental intensity data; generally these experimental $\Pi(m,p)$ values do not meet the theoretical conditions. The mismatch between theory and experiment can be used to correct the experimental data to some extent, e.g. for extinction. The corrected data then provide a better starting point for the structure determination. The assertion that semi-quantitative observed intensities contain sufficient information to be used for the direct method has not been verified. The intensity distributions of structures closely resembling one another may be so similar, that it might be impossible to distinguish between them on the basis of less accurate data.

APPENDIX

The derivation of formula (5)

Any arbitrary stacking vector $[-m/3, m/3, p/n]$, henceforth denoted as $[m,p]$, can be considered as resulting from the addition of two or more other stacking vectors provided that $p \geq 2$. For the present purpose we write each $[m,p]$ as a combination of $[\pm 1, 1] + [m \mp 1, p - 1]$. The stacking vector $[0,1]$ does not occur in silicon carbide structures (formula 2).

The allowed values of m for $p > 1$ are $0, \pm 1$, so the following combinations are therefore possible:

$$[1,p] = [0,p-1]+[1,1] \quad (a)$$
$$[1,p] = [-1,p-1]+[-1,1] \quad (b)$$
$$[0,p] = [-1,p-1]+[1,1] \quad (c)$$
$$[0,p] = [1,p-1]+[-1,1] \quad (d)$$
$$[-1,p] = [1,p-1]+[1,1] \quad (e)$$
$$[-1,p] = [0,p-1]+[-1,1] \quad (f).$$

If n_a stands for the frequency of occurrence of combination (a) etc., then

$$\Pi(1,p) = n_a + n_b$$
$$\Pi(-1,p) = n_e + n_f$$
$$\Pi(1,p-1) = n_d + n_e$$
$$\Pi(-1,p-1) = n_b + n_c.$$

Hence

$$\{\Pi(1,p)-\Pi(-1,p)\}-\{\Pi(1,p-1)-\Pi(-1,p-1)\}$$
$$=n_a+n_b-n_e-n_f-n_d-n_e+n_b+n_c$$
$$=(n_a+n_c+n_e)-(n_b+n_d+n_f) \text{ (mod. 3)}$$
$$=\{\Pi(1,1)-\Pi(-1,1)\} \text{ (mod. 3)}.$$

This is just another way of expressing formula (5), since hexagonal and trigonal SiC structures (space groups $P6_3mc$ and $P3m1$) are characterized by $\{\Pi(1,1)-\Pi(-1,1)\}=0$ (mod. 3), and rhombohedral structures ($R3m$) by $\{\Pi(1,1)-\Pi(-1,1)\}=\pm 1$ (mod.

3). The positive sign refers to the obverse setting, the negative sign to the reverse one.

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The Crystal Structure of Lanthanum Rhenium Oxide, La4Re6O19

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Lanthanum rhenium oxide, $La_4Re_6O_{19}$, is a body centered cubic phase, $a=9\cdot0325\pm0\cdot0014$ Å, space group $I23$, $Z=2$. It is formed by reacting La_2O_3, ReO_3, and Re at 1000° in vacuum. The structure contains a three-dimensional network of ReO_6 octahedra in which pairs of octahedra share an edge; the pairs in turn are linked to each other through corner sharing. The Re–Re distance in each pair is 2·42 Å, indicating metal–metal bonding. The Re–O distances range from 1·98–2·01 Å. The lanthanums are present as tetrahedral La_4O groups in open spaces of the network. The lanthanum coordination number is 10 with La–O distances of 2·50 Å (1); 2·51 Å (3); 2·60 Å (3); and 2·88 Å (3).

Introduction

If lanthanum sesquioxide, rhenium trioxide, and rhenium metal, in proportions corresponding approximately to $La_2Re_2O_7$, are heated to 1000° in vacuum, a heterogeneous product is formed (Longo, 1964). The major component of this product is a body centered cubic phase. No technique was found to separate this phase from the others present, so the stoichiometry could not be determined reliably in the normal way from density and chemical analysis. Microscopic examination of the material showed crystals apparently suitable for single-crystal study, so it was decided to solve the structure and from it establish the composition of the cubic phase.

Experimental

Longo (1964) obtained a good powder pattern for the cubic lanthanum rhenium oxide, which was indexed using a body centered cell with $a=9\cdot05$ Å. A more accurate cell length was obtained by Cohen's least-squares method (Klug & Alexander, 1954), using a

program of Leipold & Pauly (1963). The value for a obtained is $9\cdot0325\pm14$ Å.

Under the microscope a number of black (or dark blue) crystals were observed. Their habit was multi-faceted with one large face–apparently the face which had been in contact with the walls of the capsule–so that the crystals roughly approximated hemispheres. The radii of these 'hemispheres' were in the range 0·05–0·20 mm. The crystal which was selected had base diameters ranging from 0·130 to 0·165 mm and a height of 0·135 mm.

A precession camera study revealed directions with C_6 zero level symmetry and C_3 upper level symmetry as well as directions showing C_{2l} symmetry for both zero and upper levels. This establishes Laue group $m3$ (Buerger, 1942). All of the photographs showed absences for odd values of $h+k+l$. The probable space group is therefore one of $I23$, $I2_13$ and $Im3$.

The crystal was aligned with the c axis vertical when $\chi=0$ on a General Electric single crystal orienter. Zr-filtered Mo radiation was used with pulse height discrimination, a scintillation counter, and a 2° take-off angle to align the crystal and collect data. A total of 2634 reflections were measured by the moving-crystal moving-counter technique. Background was measured on each side (in θ) of each reflection. Averaging equiv-

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