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CALCULATED X-RAY DIFFRACTION DATA FOR
POLYMORPHIC FORMS OF CARBON

C. E. Holcombe

UNION
CARBIDE

OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

prepared for the U.S. ATOMIC ENERGY COMMISSION
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POLYMORPHIC FORMS OF CARBON**

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Date Issued - July 23, 1973

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ABSTRACT

Theoretical X-ray powder diffraction patterns (flat absorption) were calculated for the several forms of carbon for which crystal data are available or can be derived. These polymorphic forms (both possible or known) are either graphite related or diamond related, and are based on different stacking sequences of planes of carbon atoms.

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SUMMARY

Consideration was given to graphite-related and diamond-related forms of carbon which occur (or could occur) because of various stacking sequences of planes of carbon atoms. Crystal data were available for graphite, rhombohedral graphite, diamond, and lonsdaleite; were determined theoretically for other graphite-related forms, and were based on analogous silicon carbide or zinc sulfide structures for diamond-related forms. X-ray diffraction data for these forms of carbon were calculated and should be helpful as standard (theoretical) patterns.

INTRODUCTION

Polymorphic forms of carbon were originally thought to be confined to graphite and diamond. However, rhombohedral graphite⁽¹⁾ was detected by the presence of weak X-ray diffraction lines in addition to the usual graphite pattern. Later, Ergun and Alexander⁽²⁾ postulated a hexagonal diamond phase analogous to the 2H-SiC modification. The phase (lonsdaleite) was later prepared by Bundy and Kasper⁽³⁾ and also found to occur with meteorite diamonds. Recently, two other hexagonal forms of carbon have been found.⁽⁴⁻⁶⁾ Both are called "white carbon" and appear to be related to each other and to be similar to diamond in many properties. The first form, called chaoite, has been found mixed with normal graphite in shock-fused graphite gneisses, and has been produced at low pressures and high temperatures (free vaporization conditions) on the surface of pyrolytic graphite. The second hexagonal "white carbon" form can be produced by a laser shot on a graphite rod. This form may, in fact, not be a stable form of carbon but an intermediate form in the conversion of graphite to chaoite. Thus, at present, there appear to be six known polymorphic forms of carbon.

Gay and Gasparoux^(7,8) have reported calculated X-ray diffraction data for well-crystallized graphite (hexagonal) and rhombohedral graphite. No calculated X-ray diffraction patterns have been reported for other known carbon polymorphs. Also, there are several possible forms of carbon which have not been reported, but which theoretically could occur as distinct forms or as a result of stacking faults in a primary phase of a common type. These forms could cause the presence of weak lines which could be difficult to detect unless the patterns were known. Therefore, this study was undertaken to determine the calculated diffraction patterns of the forms of carbon for which crystal data are available or which can be derived by different stacking sequences.

CARBON X-RAY DIFFRACTION DATA

CALCULATION OF DIFFRACTION PATTERNS

Computer simulation of X-ray diffraction patterns can readily be accomplished by using a program developed by Smith.^(9,10) The program was able to correct for flat sample absorption and calculate the relative intensities.

GRAPHITE-RELATED FORMS OF CARBON

The graphite-related forms of carbon can be visualized by using the representation of rhombohedral graphite (Figure 1). If the Plane 2 of carbon atoms is removed, the stacking sequence is $[0, 1, 0]$ and is that of normal graphite. Thus, it can be seen that if the 2 plane is inserted in between any 0 or 1 plane, a series of graphite-related types would occur that have the same theoretical density as graphite but would differ in stacking sequence; and, consequently, have larger c_0 values. If the condition which occurs in rhombohedral graphite is imposed, such that each 2 plane is bounded by other planes (like 0 or 1), then the forms of graphite listed in Table 1 can occur.

The notation designating each form is the same as that used for silicon carbide forms; ie, "4H-G" as a designation shows that the form contains four atoms per unit cell, has a hexagonal (H) unit cell, and is a graphite (G)-related form of carbon. Thus, the atomic positional parameters given in Table 2 were determined from the stacking sequence of related planes of carbon atoms. The hexagonal c_0 lattice parameter for the various forms was determined by multiplying a constant (3.348 Å or 0.5 times the c_0 value for normal graphite⁽¹⁾) times the number of layers in the sequence before repeating.

The calculated diffraction patterns for graphite-related forms are contained in Table 3. It is seen that, although several additional lines occur for different forms, detection of the separate forms would be difficult. However, with the calculated patterns it is possible to differentiate between the forms. The calculated X-ray data for graphite (4H-G) and rhombohedral graphite (6R-G) compare very favorably with those of Gay and Gasparoux.^(7,8) The (006) and (104) lines for 4H-G and (009) and (107) lines for 6R-G were not included in Table 3 since their calculated intensities were < 1 .

Figure 1. RHOMBOHEDRAL GRAPHITE STRUCTURE. (To Scale)

Table 1
STACKING SEQUENCE OF PLANES OF CARBON
ATOMS FOR GRAPHITE-RELATED FORMS

Form of Carbon	Stacking Sequence ⁽¹⁾
4H-G (graphite)	[0, 1, 0]
6R-G (rhombohedral graphite)	[0, 1, 2, 0]
8H-G	[0, 2, 1, 2, 0]
10H-G	[0, 1, 2, 0, 1, 0]
12H-G	[0, 1, 2, 0, 2, 1, 0]

(1) Conditions imposed: planes designated "2" are always between those designated "0" and "1"; ie, a 2 plane, never occurs between two 0 planes or two 1 planes.

It should be noted that the calculated pattern for normal graphite (4H-G) is in general agreement (with respect to magnitude of the intensities) with that observed by Read.⁽¹¹⁾ However, the recently published diffraction file (Set 22, 1972)⁽¹²⁾ has deleted this pattern. This omission is unfortunate, since it corresponds to a very good graphite sample.

No observed X-ray diffraction pattern has been published for rhombohedral graphite (6R-G), since the purely rhombohedral form apparently cannot be isolated.

DIAMOND-RELATED FORMS OF CARBON

The forms of carbon possible which would have the same density as diamond are completely analogous to silicon carbide and zinc sulfide polytypes. The two reported tetrahedrally coordinated structures are diamond and lonsdaleite (hexagonal diamond). In diamond, the carbon layers are stacked in a cubic, close-packed array; whereas, in lonsdaleite, they are stacked in a hexagonal, close-packed array. Forming mixed cubic and hexagonal close-packed layers can be accomplished by rotating or translating carbon layers.

Figure 2 provides an alternate hexagonal representation of the stacking of carbon layers in diamond. The stacking sequences of different layers (note that "layer", as referred to here,

Table 2
ATOMIC POSITIONAL PARAMETERS FOR GRAPHITE-RELATED FORMS OF CARBON

	Form of Carbon				
	4H-G Graphite	6R-G Rhombohedral Graphite	8H-G	10H-G	12H-G
Space Group ⁽¹⁾	186	-146	186	143	187
Positions	2a:Z=0.0 2b:Z=0.0	3a:Z=0.0 3a:Z=0.3333	2a:Z=0.0 2b:Z=0.0 2b:Z=0.25 2b:Z=0.75	1a:Z=0.0 1a:Z=0.20 1a:Z=0.60 1a:Z=0.80 1b:Z=0.0 1b:Z=0.40 1b:Z=0.60 1c:Z=0.20 1c:Z=0.40 1c:Z=0.80	1a 1b 1c 1d 2g:Z=0.1667 2h:Z=0.3333 2i:Z=0.1667 2j:Z=0.3333

(1) Numbers refer to those designating space groups listed in *International Tables for X-Ray Crystallography*, 1; The Kynoch Press, Birmingham, England (1965).

Table 3
CALCULATED DIFFRACTION PATTERNS AND CRYSTAL DATA FOR
VARIOUS GRAPHITE-RELATED FORMS OF CARBON

d- Spacings (Å)	Form of Carbon ⁽¹⁾				
	4H-G Graphite	6R-G Rhombohedral Graphite	8H-G	10H-G	12H-G
<u>Intensities</u>					
3.35	100(002)	100(003)	100(004)	100(005)	100(006)
2.13	3(100)		< 1(100)	< 1(100)	
2.10			5(101)		
2.08		11(101)			6(102)
2.06				8(102)	
2.03	15(101)		12(102)		7(103)
1.99				7(013)	
1.96		9(012)			4(104)
1.80	3(102)		< 1(104)	< 1(105,015)	
1.67	5(004)	5(006)	6(008)	5(0010)	5(0012)
1.62		4(104)			2(108)
1.54	4(103)		3(106)		2(109)
1.46		2(015)			1(1010)
1.23	4(110)	4(110)	4(110)	4(110)	4(110)
1.15	6(112)	6(113,113)	6(114)	6(115,115)	6(116)
1.13	1(105)		< 1(1010)		
0.990	3(114)	3(116,116)	3(118)	3(1110,1110)	3(1112)
0.826	3(116)	3(119,119)	3(1112)	3(1115,1115)	3(1118)
0.798	1(211)		1(212)		< 1(213)
<u>Crystal Data</u>					
Theoretical Density (gms/cm ³)	2.280	2.280	2.280	2.280	2.280
Hexagonal Lattice Parameters (Å)					
a	2.456	2.456	2.456	2.456	2.456
c	6.696	10.044	13.392	16.740	20.088

(1) All lines with intensity ≥ 1 are included for graphite (4H-G) and rhombohedral graphite (6R-G). For other forms, only lines which coincide with 4H-G or 6R-G lines or for which at least one form has intensity ≥ 5 are included. Diffracting planes are designated in superscript parentheses.

means two equivalent planes of carbon atoms) can be varied to produce different forms analogous to silicon carbide polymorphs. Table 4 lists the stacking sequences along a three-fold axis for possible diamond-related forms of carbon. The notation designating each form is again analogous to that used for the silicon carbide polytypes. The positional parameters (Table 5) were those given by Wyckoff⁽¹⁾ for the silicon carbide analogs, with the silicon positions also being used for carbon atoms (except for Form 20H-D where the silicon carbide and zinc sulfide analogs differ in positional coordinates). For the 20H-D forms, both sets of positional parameters were thus used for the calculations. The space groups used in the calculations are analogous to silicon carbide. Although the true space groups may differ for pure carbon forms, this difference would not affect the calculations.

Theoretical X-ray diffraction patterns for diamond-related forms of carbon are summarized in Table 6. Lattice parameters were calculated by converting the cubic lattice parameter of diamond (3.56679 Å)⁽¹⁾ to alternate hexagonal axes, dividing the c_0 axis by the number of atoms in the alternate hexagonal cell, and multiplying this constant (1.02964 Å) by the number of atoms per cell for the other forms to obtain the value of c_0 , noting that a_0 is constant for the various forms. The theoretical pattern of diamond (8C-D) matches quite well with that listed in the Powder Diffraction File.⁽¹³⁾ The lonsdaleite (4H-D) pattern does not match very well (with respect to intensities) to that reported;⁽³⁾ however, this limitation is probably because of the presence of some cubic diamond (8C-D) which would cause distortion of the intensities because of overlapping.

As with the graphite-related forms, identification of these forms would be difficult, especially if only small amounts occurred or if they were present as ordered domains or regions of stacking faults. With the calculated patterns, however, the peaks necessary for confirmation of a particular form are apparent.

Figure 2. ALTERNATE HEXAGONAL REPRESENTATION OF DIAMOND. (To Scale)

Table 4
STACKING SEQUENCE OF LAYERS OF CARBON ATOMS
FOR DIAMOND-RELATED FORMS

Form of Carbon	Stacking Sequence
8C-D (diamond)	[0, 1, 2, 0,]
4H-D (lonsdaleite)	[0, 1, 0]
8H-D	[0, 1, 0, 2, 0]
12H-D	[0, 1, 2, 0, 2, 1, 0]
16H-D	[0, 2, 1, 2, 0, 1, 2, 1, 0]
20H-D (a) ⁽¹⁾	[0, 2, 1, 0, 1, 2, 1, 0, 1, 2, 0]
20H-D (b) ⁽²⁾	[0, 1, 2, 0, 1, 0, 2, 1, 0, 2, 0]

(1) Based on analogous SiC form.

(2) Based on analogous ZnS form.

Table 5
ATOMIC POSITIONAL PARAMETERS FOR DIAMOND-RELATED FORMS OF CARBON

	Form of Carbon						
	8C-D Diamond	4F-D Lonsdaleite	8H-D	12H-D	16H-D	20H-D (a)	20H-D (b)
Space Group ⁽¹⁾	227	194	186	186	186	156	186
Positions	8a	4f: Z=0.0625	2a: Z=0.0 2a: Z=0.1875 2b: Z=0.25 2b: Z=0.4375	2a: Z=0.0 2a: Z=0.125 2b: Z=0.1667 2b: Z=0.8333 2b: Z=0.2917 2b: Z=0.9583	2a: Z=0.0 2a: Z=0.09375 2b: Z=0.25 2b: Z=0.625 2b: Z=0.875 2b: Z=0.34375 2b: Z=0.71875 2b: Z=0.96875	1a: Z=0.0 1a: Z=0.3 1a: Z=0.7 1a: Z=0.075 1a: Z=0.375 1a: Z=0.775 1b: Z=0.2 1b: Z=0.4 1b: Z=0.6 1b: Z=0.8 1b: Z=0.275 1b: Z=0.475 1b: Z=0.675 1b: Z=0.875 1c: Z=0.1 1c: Z=0.5 1c: Z=0.9 1c: Z=0.175 1c: Z=0.575 1c: Z=0.975	2a: Z=0.0 2a: Z=0.3 2a: Z=0.075 2a: Z=0.375 2b: Z=0.1 2b: Z=0.4 2b: Z=0.7 2b: Z=0.175 2b: Z=0.475 2b: Z=0.775

(1) Numbers refer to those designating space groups listed in *International Tables for X-Ray Crystallography*, 1, The Kynoch Press, Birmingham, England (1965).

Table 6
CALCULATED DIFFRACTION PATTERNS AND CRYSTAL DATA FOR VARIOUS DIAMOND-RELATED FORMS OF CARBON

d- Spacings (Å)	Form of Carbon ⁽¹⁾						
	8C-D Diamond	4H-D Lonsdaleite	8H-D	12H-D	16H-D	20H-D (a)	20H-D (b)
				<u>Intensities</u>			
2.18		100(100)	28(100)		10(100)	8(100)	5(100)
2.17					9(101)	29(101,011)	4(101)
2.15				43(101)			
2.14						60(102,012)	26(102)
2.11			100(101)		100(102)		
2.08						100(103,013)	100(103)
2.06	100(111)	50(002)	57(004)	100(102,006)	80(008)	84(0010)	65(0010)
2.03					96(103)		
2.01						19(104,014)	56(104)
1.93		50(101)	43(102)	18(103)	14(104)	43(105,015)	2(105)
1.50		17(102)	5(104)		2(108)	1(1010,0110)	< 1(1010)
1.37				5(107)			
1.35						7(1012,0112)	3(1012)
1.32			15(105)		15(1010)		
1.28						16(1013, 0113)	16(1013)
1.26	29(220)	29(110)	33(110)	35(110,108)	44(100)	48(110)	37(110)
1.24					22(1011)		
1.22						5(1014,0114)	15(1014)
1.16		27(103)	23(106)	10(109)	8(1012)	23(1015,0115)	1(1015)
1.11						2(1016,0116)	7(1016)
1.10					8(1013)		
1.09		4(200)	1(200)	2(201)		3(202,022)	1(202)
1.08	16(311)	16(112)	18(114)	19(116)	29(118,202)	32(1110,023, 203)	20(1110,203)
1.07					6(203)	1(204,024)	3(204)
1.06		3(201)	3(202)	1(203)	1(204)	3(205,025, 1017,0117)	3(1017)

Table 6 (Continued)

d- Spacings (A)	Form of Carbon ⁽¹⁾						
	8C-D Diamond	4H-D Lonsdaleite	8H-D	12H-D	16H-D	20H-D (a)	20H-D (b)
				<u>Intensities</u>			
0.965		3(202)	< 1(204)		5(2010)		
0.910			5(205)				
0.899						6(2013,0213)	6(2013)
0.892	6(400)			5(208)			
0.882					9(2011)		
0.877						2(2014,0214)	7(2014)
0.855		15(203)	13(206)	5(209)	4(2012)	13(2015,0215)	1(2015)
0.833						2(2016,0216)	5(2016)
0.827					6(2013)		
0.826		6(210)	2(210)			< 1(210)	
0.821			8(211)		8(212)		
0.820						8(213,123)	8(213)
0.818	13(331)			10(212,2010,1014)			
0.816					9(213)		
0.815						2(214,124)	5(214)
0.809		6(211)	5(212)	2(213)	2(214)	5(215,125)	
0.806					7(1019)		
0.799						2(1024,0124)	6(1024)
				<u>Crystal Data</u>			
Theoretical Density (gms/cm ³)	3.515	3.515	3.515	3.515	3.515	3.515	3.515
Hexagonal Lattice Parameters (A)							
a	2.5221	2.5221	2.5221	2.5221	2.5221	2.5221	2.5221
c	6.1779	4.1186	8.2371	12.3557	16.4743	20.5929	20.5929

(1) All lines with intensity ≥ 1 are included for Diamond (8C-D) and Lonsdaleite (4H-D). For other forms, only lines which coincide with 8C-D or 4H-D lines or for which one form has intensity ≥ 5 are included. Diffracting planes are designated in superscript parentheses, with cubic indices for 8C-D and hexagonal indices for other forms.

CONCLUDING REMARKS

Although a large number (at least 20) of silicon carbide forms have been reported,⁽¹⁾ it is not certain whether such an extensive list would occur for graphite or diamond. However, this work shows the X-ray data for forms with a c_0 value up to about 20 Å. The computer program used^(9,10) appears to be limited to parameters of approximately 25 Å, thus preventing further calculations. Forms with larger c_0 values, if they exist, would produce more complicated diffraction patterns.

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