KNOLLS ATOMIC POWER LABORATORY
General Electric Company
Schenectady, New York

M. J. Sears

KAPL-M-LMO-4

Chemistry - General

X-RAY DIFFRACTION ANALYSIS OF BORON IN BORON CARBIDE

L. M. Osika

May 26, 1959

W. S. Horton

Authorized Classifier

5/27/59

Date

Operated for the
U. S. Atomic Energy Commission
by the
General Electric Company
Contract No. W-31-109 Eng-52

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

X-RAY DIFFRACTION ANALYSIS OF BORON IN BORON CARBIDE

L. M. Osika

ABSTRACT

By applying the relationships between lattice parameters and composition in the solid solution range, a method has been presented to analyze boron in boron carbide.

INTRODUCTION

The purpose of this method was to establish a means of determining the boron composition in a boron carbide solid solution in the presence of excess carbon or boron. The different unit-cell dimensions sometimes reported for a given solid mixture are usually attributed to the existence of solid solutions. If the crystal lattice parameters vary linearly with composition, a curve can be constructed to determine composition from lattice parameter measurements. Toward this end four samples of boron carbide of known nominal composition were selected. For each sample precision lattice parameter measurements and chemical analyses of boron and carbon content were obtained to establish relationship between lattice parameters and boron composition.

PROCEDURE

The Debye - Scherrer patterns were recorded using a Norelco asymmetric film mounting type of powder camera. The samples were powdered with a boron carbide mortar and pestle* to pass through a 325 mesh screen, and then mounted in the

* Spec Lab., 205-02 Jamaica Avenue, Hollis 23, New York

x-ray powder camera observing all the precautions necessary for precision lattice parameter determinations.⁽¹⁾ The necessary exposure time was 15 hours using Cu K α radiation and Eastman's, no screen, medical x-ray film. Except for the prohibitively long exposure time of 30 hours, Cr K α radiation would have been preferred. The films were measured to 0.05 mm directly and 0.02 mm by interpolation. The measurements were corrected for film shrinkage in the calculation of Bragg angles by the method of Straumanis.⁽²⁾ The boron carbide lines were indexed by comparing observed lines with those recorded by R. D. Allen.⁽³⁾

RESULTS AND DISCUSSION

The diffraction pattern of boron carbide with nominal composition B₄ C is tabulated in Table 1, which lists corrected (θ) in degrees for Cu K α ($\lambda = 1.5418$) pattern, the corresponding interplanar distance (d) in Angstroms, visually-estimated relative intensities and the hexagonal indices of the reflecting plane.

(1) Klug and Alexander, X-Ray Diffraction Procedures, John Wiley and Sons, pp 193-211 (1954)

(2) Ibid pp 460-1

(3) R. D. Allen, J. Am. Chem. Soc. 75 3582-3 (1953)

Table I
Boron Carbide X-Ray Diffraction Data

Cu K α			
θ°	$d \text{ \AA}^\circ$	I/I_0	hkl
9.88	4.493	30	101
11.06	4.018	40	003
11.77	3.779	70	102
16.01	2.795	30	110
17.54	2.558	80	104
18.94	2.375	100	201
19.59	2.299	10	113
25.22	1.809	5	211
26.83	1.708	30	205
28.43	1.619	5	107
30.95	1.499	20	303
31.90	1.459	30	125
32.37	1.447	30	108
33.41	1.400	30	220
35.24	1.336	20	311
35.68	1.322	20	223
35.97	1.312	20	312
37.03	1.280	10	208
37.86	1.256	20	217
39.89	1.202	5	401
40.43	1.1887	10	402
41.42	1.1652	10	218
42.18	1.1481	10	226

Lattice parameters were obtained by calculating the a_0 and c_0 from high angle lines with low and high indices respectively. Table two lists the x-ray lattice parameters calculated and the boron chemical analyses for the four nominal compositions $B_{3.7}C$, B_4C , $B_{4.5}C$ and B_6C . An emission spectrographic analyses of the four samples showed about 1% impurities — mostly iron.

Table 2

Nominal Composition	X-Ray		Chemical Analysis	
	a_0 $\pm 0.005 \text{ \AA}$	c_0 ± 0.01	B w/o	B/C Ratio
$B_{3.7}C$	5.677	12.76	74.5	$B_{3.5}C$
B_4C	5.678	12.78	75.8	$B_{3.6}C$
$B_{4.5}C$	5.625	12.11	78.5	$B_{4.3}C$
B_6C	5.645	12.15	82.11	$B_{5.7}C$

This data was used to construct the boron composition versus lattice parameter curve (Figure 1) which was used in all subsequent boron in boron carbide determinations by x-ray diffraction.

COMMENTS

Because it was necessary to use Cu K α radiation to avoid longer x-ray exposure times, there was a lack of resolution in the back reflection lines. This did not permit an unambiguous indexing of the back reflection, therefore, the use of an extrapolation method for precision lattice parameter measurements could not be used. Instead the precision indicated in lattice parameter

measurements was attained by applying a good sample preparation technique to obtain x-ray patterns which could be measured with high precision. This was done by controlling the following conditions; fine powder size to improve character of x-ray diffraction lines, small sample size to minimize the absorption error and the use of an asymmetric film loaded camera. Since each film is self calibrated, allowance is automatically made for differences in film shrinkage resulting from variations in film processing conditions.

The lower solubility limit of the solid solution of crystalline carbon in boron carbide corresponds to $B_{3.4}C$ with lattice parameters of $a_0 = 5.598 \pm 0.005 \text{ \AA}$ and $c_0 = 12.06 \pm 0.01 \text{ \AA}$. The lattice parameters of boron carbide in equilibrium with crystalline boron have tentatively been established as $a_0 = 5.652 \pm 0.005 \text{ \AA}$ and $c_0 = 12.16 \pm 0.01 \text{ \AA}$ which corresponds to $B_{5.9}C$.

FIGURE 1

150x250 mm Divisions

GENERAL ELECTRIC COMPANY, SCHENECTADY, N. Y., U. S. A.

FN-156 (12.49)