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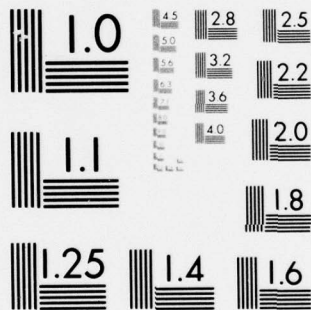
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# SPECTROCHEMICAL ANALYSIS OF BORON CARBIDE BY POWDER TECHNIQUES

BERNARD H. STRAUSS and FRED G. HOWELL  
POLYMER AND CHEMISTRY DIVISION

February 1977



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ABSTRACT

A method for the determination of impurities in boron carbide was developed using powder techniques. The elements aluminum, calcium, iron, magnesium, manganese, nickel, silicon, titanium, and vanadium were determined over concentration ranges between 10 and 24,000 parts per million. The boron carbide powder was mixed with buffers including an internal standard. These sample mixtures were excited in a direct current arc and spectra recorded on photographic plates. Due to a lack of standard boron carbide samples, the analytical curves are based on the wet chemical analysis of six samples. Dilution with graphite was employed to extend the analytical curves of the higher level elements (aluminum, calcium, iron, and silicon) down to 100 ppm or less.

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## INTRODUCTION

Boron carbide is a refractory material usually produced at temperatures above 1500 C. At 1000 C to 1100 C<sup>1</sup> it reacts with metals such as aluminum, calcium, chromium, iron, magnesium, nickel, silicon, titanium, and vanadium to form borides or carbides. Commercial boron carbide is generally of low purity with concentration ranges of the above elements varying from 5 to 24,000 parts per million.

A study by Matchen<sup>2</sup> showed that the ballistic properties of boron carbide improve in relation to a decrease in its impurity levels. However, the controlled addition of small amounts of certain impurities can enhance specific properties of high-purity materials, for example, by minimizing recrystallization or by allowing full densification, but the uncontrolled presence of undefined impurities cannot be expected to have any beneficial effect.

Chemical analysis of boron carbide is generally performed by classical "wet" chemical techniques; however, almost all such procedures are very time consuming. Accordingly, in this laboratory, spectrographic techniques were investigated and methods were developed which require little sample preparation. These methods require standards and none are commercially available. Therefore, boron carbide samples were obtained from various sources and analyzed by classical chemical techniques for the purpose of this study. A report by Ferraro and Czyrkliś describes the methods used for the determination of iron, titanium, aluminum, silicon, and nickel.<sup>3</sup> Ferraro\* has since developed methods for the determination of calcium, manganese, magnesium, and vanadium. The analysis of the six samples used as standards for this method are listed in Table 1.

## EXPERIMENTAL

The analyzed samples consisted of coarse powders which were ground to 200 mesh size or smaller to facilitate intimate mixing with spectrographic buffers. A steel mortar and pestle were used to achieve this and a magnet was passed through the fine powder many times to insure complete removal of adventitious iron.

Direct excitation of undiluted boron carbide powder was attempted, but the spectral lines were buried in a high background and line intensities could not be measured. The use of buffers mixed with the material of interest

Table 1. IMPURITIES IN BORON CARBIDE SAMPLES AS DETERMINED BY WET ANALYSIS METHODS (in parts per million by weight)

| Element | Sample Number |       |       |     |        |        |
|---------|---------------|-------|-------|-----|--------|--------|
|         | E-1           | E-2   | E-3   | E-4 | E-5    | E-6    |
| Al      | 2,970         | 3,070 | 4,015 | 431 | 532    | 2,145  |
| Ca      | 285           | 504   | 1,030 | 900 | 2,790  | 1,000  |
| Fe      | 3,145         | 2,295 | 2,195 | 751 | 23,950 | 3,990  |
| Mg      | 23            | 30    | 32    | 11  | 92     | 1,000* |
| Mn      | 26            | 31    | 26    | 5   | 190    | 890*   |
| Ni      | 108           | 116   | 91    | 39  | 250    | 75     |
| Si      | 870           | 615   | 739   | 339 | 664    | 1,560  |
| Ti      | 1,200         | 255   | 514   | 637 | 93     | 63     |
| V       | 46            | 175   | 445   | 39  | 993    | 81     |

\*Insufficient sample for spectrographic use.

1. ALEXANDER, M. N. *Boron Carbide - A Survey of Solid-State Properties*. Army Materials and Mechanics Research Center, AMMRC TR 71-7, May 1971.
2. MATCHEN, B. *Development of Improved Ceramic Armor Materials*. Norton Research Corporation, Ltd., Cheppowa, Ontario, Contract DAAG46-68-C-0096, Confidential Report, AMMRC CR 70-21, January 1971, AD 515-580L.
3. CZYRKLIŚ, W. D., and FERRARO, T. A. *Photometric Methods for the Determination of Iron, Titanium, Aluminum, Silicon and Nickel in Boron Carbide*. Army Materials and Mechanics Research Center, AMMRC PTR 71-1, July 1971.

\*FERRARO, T. A., private communication.

is one method of reducing background, so various buffers were tried including graphite with 20% silver chloride and graphite with 10% and 20% iron oxide. These did not prove satisfactory as poor correlation was obtained between density readings and concentration values on the analytical curves. Dilution of boron carbide with magnesium oxide also gave poor results. The magnesium oxide-boron carbide mixture packed so tightly in the electrode that, even with venting, blowout could not be controlled.

A procedure that gave satisfactory results included the mixing of boron carbide with such buffers as graphite, lithium carbonate, or lithium fluoride. Cobalt oxide was also added in constant amounts as an internal standard so that elemental lines of interest could be referenced against certain cobalt lines. No cobalt was found in any of the analyzed samples and there were sufficient cobalt lines available to choose good analytical line pairs with the various elements being investigated. Graphite was used to establish a uniform matrix and also as a diluting agent. The lithium fluoride, lithium carbonate, and cobalt oxide were of high purity and obtained from SPEX Industries. The lithium carbonate buffer, called Buffer A, was utilized for the determination of aluminum, calcium, and silicon. It consisted of 55% graphite, 44% lithium carbonate, and 1% cobalt oxide. These three powders were mixed in plastic vials using a plastic ball pestle and the contents shaken in a SPEX mixer/mill for five minutes. Boron carbide was then mixed in proportions of one to nine with Buffer A in a plastic vial and the contents were also shaken for five minutes.

Buffer B, used for the remaining elements and also applicable to calcium, consisted of 78% graphite, 20% lithium fluoride, and 2% cobalt oxide. These powders were mixed as described above. One part boron carbide was then mixed with one part Buffer B. Table 2 describes the sample preparations used with Buffers A and B.

Thirty milligram samples of the boron carbide-buffer mixtures were loaded into deep-cupped 3/16-inch-diameter graphite electrodes. The electrodes were heated in an oven at 120 C overnight or for a few hours to remove any absorbed

Table 2. PREPARATION OF SAMPLE-BUFFER MIXTURES

---

|                                                                    |
|--------------------------------------------------------------------|
| I. Determination of Al, Ca, and Si.                                |
| A. Buffer A                                                        |
| 1. 2.45 grams of 200 mesh graphite                                 |
| 2. 2.00 grams of lithium carbonate                                 |
| 3. 0.05 gram of cobalt oxide                                       |
| B. Mix 30 mg of B <sub>4</sub> C sample and 270 mg of Buffer A.    |
| C. Weigh 30 mg of sample into a 3/16"-diameter graphite electrode. |
| II. Determination of Mg, Mn, Fe, Ni, Ti, Ca, and V.                |
| A. Buffer B                                                        |
| 1. 3.51 grams of 200 mesh graphite                                 |
| 2. 0.90 gram of lithium fluoride                                   |
| 3. 0.09 gram of cobalt oxide                                       |
| B. Mix 100 mg of B <sub>4</sub> C sample and 100 mg of Buffer B.   |
| C. Weigh 30 mg of sample into a 3/16"-diameter graphite electrode. |

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water vapor. The electrodes were then mounted in a water-cooled Stallwood<sup>4</sup> jet through which a 6-SCFH (standard cubic feet per hour) flow of a mixture of 80% argon and 20% oxygen was passed. This gas mixture was used to prevent the formation of interfering cyanogen bands. To counter the loss of material at the time of ignition, a very low initial amperage (2 to 3 amperes for a few seconds) was used. This caused less violent action and fused the surface of the sample.

Extended exposures of up to 60 seconds were used to minimize the effect of fluctuations in burning characteristics during the exposure. Neutral density filters were employed, where necessary, to decrease high intensities to measurable levels. Operating parameters are shown in Table 3.

The sample electrode was ASTM S-14 and the counter electrode was Ultra Carbon 105-U. Eastman SA-1 photographic emulsion on glass plates was developed using Kodak D-19. A Baird 3-meter concave grating with 600-groove/mm, Eagle mount, and a reciprocal linear dispersion of 2.75 Å/mm in the second order was employed for this work. The microphotometer was a Jarrell-Ash nonrecording Model 2100.

A study was made to determine the feasibility of employing graphite dilution to extend the analytical curves of the higher level elements, namely aluminum, calcium, iron, and silicon, down to 100 ppm or less. The procedure involved serial dilutions with graphite to obtain the desired concentrations. The diluted samples were then mixed in equal proportions with Buffer B. Portions of these mixtures were finally further diluted tenfold with graphite. This was done to maintain a fairly constant level of graphite in the final samples. Table 4 illustrates this by the example of sample E-6 which was initially diluted by factors of 2 to 64 while the final graphite content varies less than five percent.

Table 3. OPERATING PARAMETERS\*

| Element Sought | Buffer Mixture | Wavelength Range (Å)   | Current (amp) | Pre-Arc (sec) | Exposure (sec) | Filters (% Transmission) |
|----------------|----------------|------------------------|---------------|---------------|----------------|--------------------------|
| Al, Ca, Si     | A              | 2750-3450<br>2nd Order | 12            | 0             | 40             | 20/4<br>Masked to 3 mm   |
| Mg, Mn         | B              | 2405-3105<br>2nd Order | 14            | 0             | 40             | None                     |
| Fe, Ni, Ti     | B              | 2750-3450<br>2nd Order | 12            | 0             | 30             | None                     |
| Ca, V          |                | 2750-3450<br>2nd Order | 20            | 20            | 60             | 100/20/4                 |

\*Sample excitation performed in a Stallwood jet with an 80% argon - 20% oxygen mixture at a flow rate of 6 SCFH.

Table 4. COMPOSITION OF SAMPLE E-6

| Initial Dilution            |                     |                                    | After 1:9 Dilution with Graphite |               | Difference of Graphite Content Between Dilutions (%) |
|-----------------------------|---------------------|------------------------------------|----------------------------------|---------------|------------------------------------------------------|
| Wt of B <sub>4</sub> C (mg) | Wt of Graphite (mg) | Ratio of B <sub>4</sub> C:Graphite | % of B <sub>4</sub> C            | % of Graphite |                                                      |
| 75.0                        | 75.0                | 1:1                                | 5.0                              | 95.0          | -                                                    |
| 37.5                        | 112.5               | 1:3                                | 2.5                              | 97.5          | 2.5                                                  |
| 18.8                        | 131.3               | 1:7                                | 1.3                              | 98.8          | 3.8                                                  |
| 9.4                         | 140.7               | 1:15                               | 0.6                              | 99.4          | 4.4                                                  |
| 4.7                         | 145.4               | 1:31                               | 0.3                              | 99.7          | 4.7                                                  |
| 2.3                         | 147.4               | 1:63                               | 0.2                              | 99.8          | 4.8                                                  |

4. STALLWOOD, B. J. *Air-Cooled Electrodes for the Spectrochemical Analysis of Powders*. Journal of Optical Society of America, v. 44, 1954, p. 171-176.

The ranges of concentration for aluminum, calcium, iron, and silicon were extended as shown in Table 5. For example, the lowest value of aluminum in the standards was 431 ppm. This was extended down to 10 ppm. Calcium was decreased in concentration from 285 to 20 ppm, iron from 751 to 100 ppm, and silicon from 339 to 10 ppm. Figure 1 illustrates an analytical curve for iron composed of samples E-2 and E-4 and their dilutions. Note that no dilution effect is apparent.

Typical analytical curves using both types of buffers for the various elements are shown in Figure 2.

Table 5. DILUTED SAMPLE RANGES FOR VARIOUS ELEMENTS

| Element | Concentration Range (ppm) |
|---------|---------------------------|
| Al      | 10 to 4,015               |
| Ca      | 20 to 2,790               |
| Fe      | 100 to 23,950             |
| Si      | 10 to 1,560               |

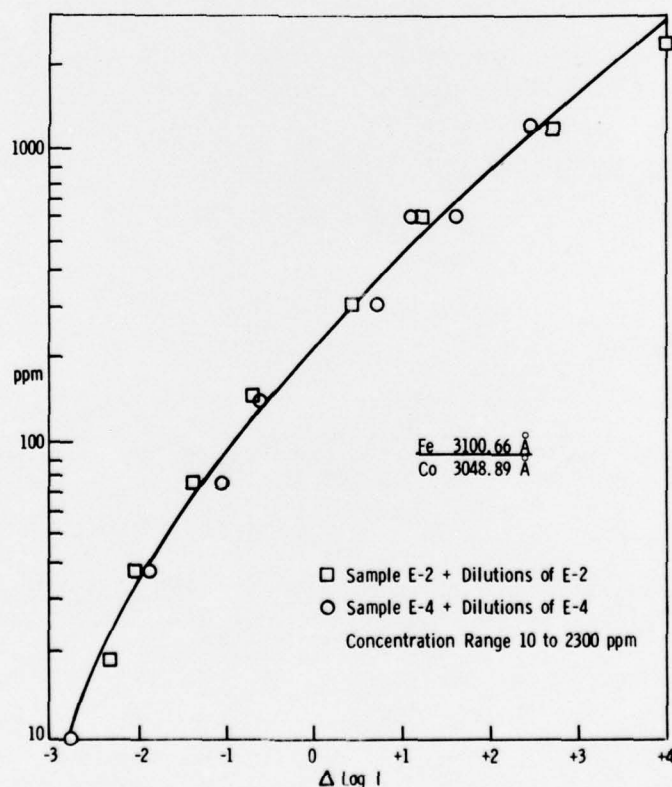


Figure 1. An analytical curve for iron composed of samples E-2 and E-4 and their dilutions.

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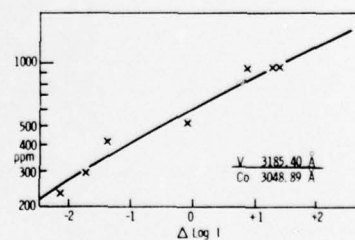
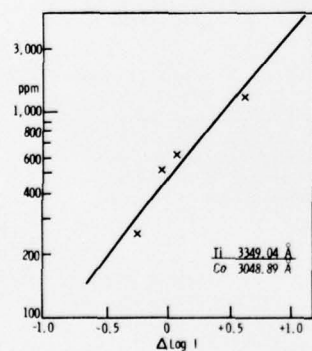
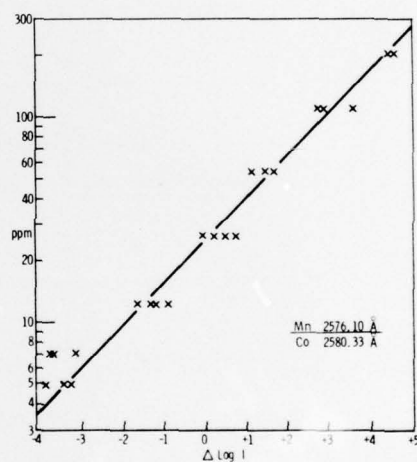
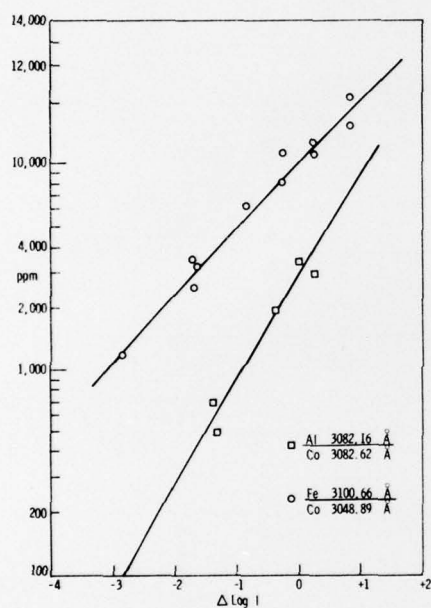
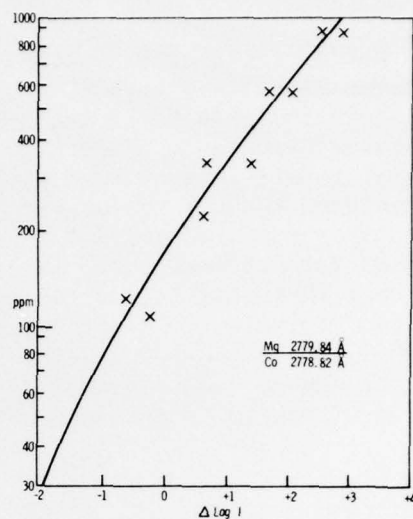
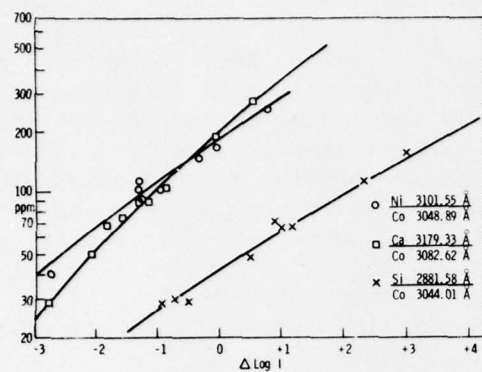


Figure 2. Typical analytical curves for various elements.

## RESULTS AND DISCUSSION

Coefficients of variations were calculated as outlined in "Methods for Emission Spectrochemical Analysis."<sup>5</sup> The statistical results on the nine elements determined are shown in Table 6. Points on the analytical curves correspond to concentration of these elements in either the standard samples or a mixture of standards. Three to five repetitions of each point were taken, depending on the number of exposures possible on the spectrographic plate. If no filters were needed, and with a line mask of approximately 1.5 mm, 36 exposures were possible. The coefficients of variation for calcium, iron, manganese, nickel, and vanadium were generally lower than 15% over their working curves in the range of concentration as listed in Table 1. The coefficients for aluminum, magnesium, and titanium were generally about 25%; however, the magnesium concentration range is only 10 to 100 ppm. Silicon values did not exceed 9.0%. Insufficient sample E-6 was available to use for the determination of magnesium and manganese as shown in Table 1.

Table 6. ANALYTICAL PRECISION OF DATA

| Element | Sample Number | Working Curve Points (ppm) | No. of Curves Data Taken From | Coefficient of Variation |
|---------|---------------|----------------------------|-------------------------------|--------------------------|
| Al      | E-4           | 431                        | 11                            | 22.7                     |
|         | E-6           | 2,145                      | 9                             | 23.8                     |
|         | E-2           | 3,070                      | 14                            | 18.2                     |
| Ca      | E-1           | 285                        | 3                             | 24.1                     |
|         | E-4           | 900                        | 8                             | 10.8                     |
|         | (E-5 + E-4)/2 | 1,845                      | 5                             | 3.5                      |
|         | E-5           | 2,790                      | 8                             | 11.5                     |
| Fe      | E-4           | 751                        | 4                             | 8.6                      |
|         | E-3           | 2,195                      | 7                             | 8.0                      |
|         | (E-4 + E-5)/2 | 12,350                     | 4                             | 23.5                     |
|         | E-5           | 23,950                     | 7                             | 14.0                     |
| Mg      | E-4           | 11                         | 5                             | 27.9                     |
|         | E-3           | 32                         | 3                             | 24.2                     |
|         | E-5           | 92                         | 6                             | 12.3                     |
| Mn      | E-1           | 26                         | 4                             | 14.1                     |
|         | E-5           | 190                        | 4                             | 8.2                      |
| Ni      | E-4           | 39                         | 6                             | 6.9                      |
|         | E-3           | 91                         | 6                             | 12.6                     |
|         | (E-4 + E-5)/2 | 145                        | 4                             | 15.4                     |
|         | E-5           | 250                        | 6                             | 4.7                      |
| Si      | E-4           | 339                        | 5                             | 4.3                      |
|         | E-5           | 664                        | 11                            | 0.8                      |
|         | E-1           | 870                        | 12                            | 8.2                      |
|         | E-6           | 1,560                      | 6                             | 3.2                      |
| Ti      | E-2           | 255                        | 7                             | 27.5                     |
|         | E-3           | 514                        | 5                             | 42.7                     |
|         | E-1           | 1,200                      | 7                             | 17.4                     |
| V       | E-2           | 175                        | 3                             | 12.1                     |
|         | (E-5 + E-6)/2 | 537                        | 4                             | 9.9                      |
|         | E-5           | 993                        | 4                             | 6.7                      |

5. *Methods for Emission Spectrochemical Analysis*. American Society for Testing and Materials, 6th edition, 1971, p. 383-386.

The selected analytical line pairs, listed in Table 7, are free of interferences except that of vanadium 3082.11 Å on aluminum 3082.16 Å and titanium 3100.67 Å on iron 3100.66 Å. According to Meggers,<sup>6</sup> the vanadium line is 1/16 the intensity of the aluminum line. Thus, with a vanadium concentration twice that of the aluminum, no measurable effect was observed. Titanium at 3100.67 Å with a Meggers' intensity of 15 did not affect the iron 3100.66 Å line with an intensity of 26 at a titanium concentration 1-1/2 times greater than that of iron.

This proposed method enables one to rapidly analyze boron carbide powder with high sensitivity. One is able to determine aluminum at 10 ppm, iron at 100 ppm, and silicon at 10 ppm, as compared to spectrographic solution work done in this laboratory where the limits of detection for aluminum, iron, and silicon were 230, 774, and 287 ppm, respectively, although with improved coefficients of variation of approximately five percent.

|    | Analytical Line<br>(Å) | Cobalt Internal<br>Standard Line (Å) |
|----|------------------------|--------------------------------------|
| Al | 3082.16                | 3082.62                              |
| Ca | 3179.33                | 3082.62                              |
| Fe | 3100.66                | 3048.89                              |
| Mg | 2779.84                | 2778.82                              |
| Mn | 2576.10                | 2580.33                              |
| Ni | 3101.55                | 3048.89                              |
| Si | 2881.58                | 3044.01                              |
| Ti | 3349.04                | 3048.89                              |
| V  | 3185.40                | 3048.89                              |

#### RECOMMENDATIONS

It is recommended that the powder-spectrographic method proposed in this report, details of which are presented in Tables 2 and 3, be included in Army purchase descriptions of materials and products composed of boron carbide.

6. MEGGERS, W. F., CARLIOS, C. H., and SCRIBNER, B. F. *Tables of Spectral-Line Intensities, Part I*. NBS Monograph 32, U.S. Printing Office, December 1961.

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