Article

Determination of Boron in Silicon Carbide by ICP-OES and ICP-MS after Sample Preparation using Pyrohydrolysis

Cristiano C. Muller, Thieli S. Nunes, Aline L. H. Muller, Valderi L. Dressler, Erico M. M. Flores and Edson I. Muller*

Departamento de Química, Universidade Federal de Santa Maria, 97105-990 Santa Maria-RS, Brazil

A pirohidrólise foi utilizada no preparo de amostras de carbeto de silício (SiC) para posterior determinação de B usando espectrometria de emissão óptica com plasma indutivamente acoplado (ICP-OES) e espectrometria de massa com plasma indutivamente acoplado (ICP-MS). Parâmetros importantes como temperatura, fluxo de água e ar, tempo de reação e uso de V_2O_5 foram avaliados. Adicionalmente, foi avaliada a interferência de W sobre as linhas de emissão de B durante as determinações por ICP-OES. A exatidão foi avaliada usando material de referência certificado (CRM BAM S003) e valor de bias inferior a 1% foi encontrado entre os valores de referência e obtido experimentalmente. Limite de detecção (LOD) de 0,010 µg g^{-1} foi obtido usando pirohidrólise e determinação por ICP-MS. A pirohidrólise é um método de preparo de amostras simples que permitiu a utilização de água deionizada como solução absorvedora para a determinação de B em SiC.

Pyrohydrolysis was used for sample preparation of silicon carbide (SiC) and subsequent determination of B using inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS). Important parameters for pyrohydrolysis method as temperature, air and water flow rates, reaction time and use of V_2O_5 were evaluated. In addition, a study of W spectral interference on B emission lines was evaluated for ICP-OES determination. Accuracy was evaluated using certified reference material (CRM BAM S003) and bias value lower than 1% between the certified and determined value was achieved. Limit of detection (LOD) of 0.010 µg g^{-1} was obtained using pyrohydrolysis and ICP-MS. Pyrohydrolysis is a very simple sample preparation method that allowed the use of deionized water as absorbing solution for B determination in SiC.

Keywords: silicon carbide, pyrohydrolysis, boron, ICP-MS, ICP-OES

Introduction

Silicon carbide (SiC) is an advanced ceramic material used in the semiconductor industry. In addition, it has a great chemical resistance and can also be used under extreme temperature conditions.[1-3] The electrical properties of SiC can be changed by adding dopants agents. Boron is a doping agent widely used for controlling the electronic properties of SiC and other semiconductor materials. For example, doping of B in SiC increases the electrical conductivity of this material from ca. 10^{-12} to ca. 10^{-3} Ω^{-1} cm^{-1} and decreases the band gap from 2.0 to 1.7 eV.[4,5] Additionally, B may also be present as a contaminant arising from the materials used in the synthesis of silicon carbide.[6]

Therefore, there is a need for adequate accurate methods for B determination in this material. The main digestion methods for decomposition of SiC have been performed by closed vessels digestion with a concentrated acids mixture and fusion with different reagents.[7-11] SiC is a refractory material difficult to bring into solution and closed vessels digestion in poly(tetrafluoroethylene) (PTFE) bomb with concentrated HNO_3, H_2SO_4 and HF mixture and heated up to 20 h for complete sample decomposition is required. The limit of detection (LOD) in ppm range can be achieved for B using wet digestion methods.[7-11] The main drawbacks related to wet digestion of SiC is the possibility of contamination, losses of volatile species and incomplete dissolution of sample. A fusion with alkaline mixtures of NaOH or Na_2CO_3 and Na_2O_2 is widely used. However, the high salt content of final fusion digests requires the dilution

*e-mail: edson_muller@yahoo.com.br

of these solutions, consequently worse limits of detection are obtained. Moreover, contamination by the reagents and environment can occur during the fusion procedure.[8] Determination of impurities in SiC can also be carried out without an extensive sample preparation step. In this sense, a direct introduction of SiC samples using an inductively coupled plasma optical emission technique (ICP-OES), such as, direct slurry nebulization and electrothermal vaporization (ETV), can be carried out.[12-16] Direct slurry nebulization in ICP-OES is applied for trace element determination in SiC samples and enables an improvement in LOD in comparison with methods that require previous digestion of sample. Limitations related to particle size (below 10 μm level) and slurry stability are observed for this technique.[16] A sample introduction in the ICP-OES by ETV device is applied for SiC quality control,[17] but for B determination by ETV-ICP-OES, chemical modifiers are required in order to improve the B sensitivity.[18,19] A direct solid sampling electrothermal atomic absorption spectrometry (DSS-ETAAS) is also used for trace element determination of ceramics materials.[20,21] However, due to poor sensitivity of B using ETAAS, it is difficult to determine B at trace level in SiC samples.[22] Direct current arc (DC arc)[16] and spark ablation,[23] both using optical emission spectrometry for detection, also allow the direct analysis of SiC samples. However, with these techniques, good accuracy can only be obtained by using solid material standards with similar matrix for calibration.[24]

Pyrohydrolysis is an alternative method for sample preparation and allows the vaporization of the analyte by hydrolysis under high temperature (around 1000 °C) with substantial reduction of interference of the matrix for determination using plasma techniques.[25,26] Instrumentation required for a pyrohydrolysis analysis is very simple, minimal sample preparation is required, a sample mass in gram range can be used and the absorbing of pyrohydrolysis products is carried out using dilute solutions of alkalis or acids depending on the characteristics of analyte.[27] In this sense, pyrohydrolysis is suitable for a refractory ceramic analysis, due to the low LOD and minimum interference of the matrix in the determination technique.[28-31] Numerous studies devoted to halogens determination in different samples are found in the literature.[25,26,31-39] However, pyrohydrolysis can be used for determination of other elements such as S and B.[27-30,40,41] Volatilization of B compounds during pyrohydrolysis is probably explained by the formation of gaseous HBO_2 or H_3BO_3.[30] Sometimes, the use of auxiliary reagents (such as, V_2O_5) is required in order to assure the complete recovery of analytes during the pyrohydrolysis procedure.[41] In this work, a pyrohydrolysis method was proposed for high purity SiC powder digestion and further B determination by ICP-OES and ICP-MS. Important pyrohydrolysis parameters, such as, air and water flow rates, temperature, reaction time, sample mass and use of catalyst were evaluated. In addition, the main spectral interference caused by tungsten emission lines on B determination using ICP-OES was also studied.

Experimental

Reagents, solutions and samples

All chemical reagents used were of analytical grade and purchased from Merck (Darmstadt, Germany). Nitric acid (65% m/m) was doubly distilled in quartz sub-boiling still (Milestone, Sorisole, Italy, model duoPUR 2.01E). Sulfuric and hydrofluoric acids were of "Suprapur" grade (Merck, Darmstadt, Germany). Water used for all procedures was distilled, deionized, and further purified in a Milli-Q system (18.2 MΩ cm, Millipore Corp., Bedford, USA). Standard solutions for calibration of B, V and W were prepared by diluting the stock solutions of 1000 mg L^{-1} (all from SpexCerti Prep, Metuchen, NJ). Accelerator agent, V_2O_5, was obtained by heating NH_4VO_3 in a platinum crucible at 550 °C on furnace for 4 h. Certified reference material (CRM) of silicon carbide powder BAMS003 (Federal Institute for Materials Research and Testing - BAMS003) was analyzed for method accuracy evaluation. Four SiC samples used for analysis were donated by a Brazilian industry. SiC samples were not milled and they were used as received from the industry (particle size lower than 80 μm).

Instrumentation

Boron determination was carried out using an inductively coupled plasma optical emission spectrometer (Ciros CCD model, Spectro, Kleve, Germany) equipped with a Scott-type double pass spray chamber (glass material), cross-flow nebulizer (glass material), torch and injector of quartz. The operation conditions were: radio frequency power of 1.6 kW, principal plasma gas flow rate of 14.0 L min^{-1}, auxiliary gas flow rate of 1.0 L min^{-1} and nebulizer gas flow rate of 0.80 L min^{-1}. For all measurements, the axial view and wavelengths of B I 249.677 nm, B I 249.772 nm, B I 208.889 nm and B I 182.639 nm emission lines were used. Determination of B was also performed using inductively coupled plasma mass spectrometer (Elan DRC II model, Perkin Elmer, Thornhill, Canada) equipped with concentric nebulizer and cyclonic spray chamber. Radio frequency power of 1.3 kW, principal plasma gas flow rate of 15.0 L min^{-1},

auxiliary gas flow rate of 1.2 L min^{-1}, nebulizer gas flow rate of 1.05 L min^{-1} and isotope (*m/z*) of ^{11}B were used as operation conditions. Concentric nebulizer (glass material), baffled cyclonic spray chamber (glass material), torch (quartz material) and injector (quartz material) were used as ICP component for sample introduction.

Microwave-assisted acid digestion of SiC samples was carried out in microwave oven (Multiwave 3000 model, Anton Paar, Austria) using PTFE-TFM vessels.

Sample preparation using microwave-assisted acid digestion

Silicon carbide masses up to 100 mg were transferred to PTFE-TFM vessels and 6 mL of a mixture of concentrate nitric, sulfuric and hydrofluoric acids (2 mL of each acid) was mixed together. PTFE-TFM vessels were transferred inside the microwave oven and the heating program applied was (*i*) 25 °C to 230 °C ramp of 20 min, (*ii*) 230 °C during 120 min, and (*iii*) 230 °C to 25 °C ramp of 20 min (cooling step). Samples were diluted up to 25 mL with deionized water. In order to minimize deleterious effects of HF on quartz torch, samples were diluted (5 fold) before analysis by ICP-OES and ICP-MS. These B results were used to evaluate the B recovery using the proposed pyrohydrolysis method.

Pyrohydrolysis apparatus

The pyrohydrolysis system used in this work is described in a previously published study.[26] Air flow was supplied by an air compressor and a control of flow rate was performed using a fluxometer (2A13 model, Key Instruments, USA). Deionized water was used for the pyrohydrolysis reaction and was pumped using a peristaltic pump (IPC8 ISM931 model, Ismatec, Swiss). Air and water introduction into the high purity fused silica reactor tube (35 cm of length, 1.6 cm of i.d. and 1.9 cm e.d.) were performed using two alumina capillaries. Water was pumped through the heated alumina capillary and converted to vapor after introduction inside the heated reactor tube. A silicon carbide sample was placed inside an alumina boat and introduced inside the reactor. The reactor was heated using a resistive furnace with a maximum temperature of 1100 °C (Sanchis, Porto Alegre, Brazil). The outlet of the reactor was connected to a serpentine, which was immersed in an ice bath to condense the pyrohydrolysis products. The condensed products were collected in a vessel containing 10 mL of deionized water and diluted up to 50 mL in volumetric flask. Boron determination was carried out in this solution without dilution.

Pyrohydrolysis procedure

Initial tests for pyrohydrolysis optimization parameters were carried out weighing 200 mg of SiC in an alumina boat to evaluate the air flow rate during the sample heating. In this sense, 0.3 L min^{-1} of air was used in combination with 1.0 mL min^{-1} of water pumped through heated capillary. Reaction time and temperature of 10 min and 1000 °C were used, respectively. Water (10 mL) was used to absorb the pyrohydrolysis products. In order to improve the B recovery, important pyrohydrolysis parameters were evaluated: air flow rate (ranging from 0.1 to 1.0 L min^{-1}), water flow rate pumped through heated capillary (ranging from 0.25 to 2.5 mL min^{-1}), temperature (750 °C to 1100 °C), auxiliary reagent (without V$_2$O$_5$ and maximum of 800 mg of V$_2$O$_5$) and reaction time (7.5 min up to 25 min). Prior to each analysis, the alumina boat was soaked with a nitric acid solution (1 + 1) for 15 min, and then rinsed with water and dried. After the pyrohydrolysis of each sample, the system was heated for another 10 min to assure cleaning of the system. The visual aspect of SiC powder remains unchanged after reaction, indicating that no chemical reaction occurred during pyrohydrolysis.

Results and Discussion

A previous analysis of SiC wet digestion solutions showed important spectral interferences of tungsten on B main emission lines. In this sense, a study of W interferences in analysis of digest solution obtained by wet digestion and standard solutions containing vanadium and W was carried out. Only the secondary emission lines were completely free of interferences and could be used for B determination. Isobaric or polyatomic interferences were not observed in ^{11}B determination by ICP-MS and this isotope was used for the analysis of SiC wet digestion solutions.

Silicon carbide samples and CRM (BAM S003) were also digested using microwave-assisted acid digestion and results for B are shown in Table 1. SiC I and SiC II samples showed the highest values for B, while sample SiC III e Si IV had B levels lower than the limit of detection obtained by ICP-MS (lower than 0.01 µg g^{-1}).

Alkaline solutions[41] or only deionized water[30] are usually used for B absorption after pyrohydrolysis volatilization. In this sense, diluted alkaline solutions 0.1 mol L^{-1} of NaOH and deionized water were evaluated for B absorbing. Significant differences were not observed and deionized water was selected due to minimum interference in nebulization system and plasma stability.

Tungsten spectral interferences were not observed in analysis of wet digestion and pyrohdrolysis digests of CRM (silicon carbide, BAM S003) using ICP-OES.

SiC II sample was available in larger amounts and it was used to optimize the pyrohydrolysis parameters. In this sense, the best conditions in the parameters evaluated for pyrohydrolysis were selected using the highest recovery of B for SiC II sample. After optimizing all the important parameters, the determination of B in the certified reference material was carried out in order to evaluate the accuracy of the proposed method.

Finally, it is also important to mention that preliminary tests were also performed in order to evaluate presence of halogens (F, Cl, Br and I) in SiC samples. However, these elements were below LOD when ICP-MS and ion chromatography were used and the method was not optimized for determination of these elements.

Spectral interferences

During optimization of the pyrohydrolysis method, spectral interferences for main B emission lines[42] at B I 249.677 and B I 249.772 nm were observed. Consulting the spectral database of NIST, tungsten has more than five thousand emission lines between 180 and 300 nm.[42] Probably, the interference is caused by tungsten emission lines, because SiC samples can be ground using a tungsten carbide mill during industrial process. In this sense, carbide tungsten contamination can occur during milling of SiC samples.[43] Unfortunately, information regarding to industrial process of SiC samples used in this study was not provided by the industry. Volatilization of tungsten during pyrohydrolysis occurs due to probable formation of hydrated tungsten oxides with high volatility.[44] In Figure 1 it is possible to observe the signals for B I 249.677 nm and B I 249.772 nm (main lines for B) for blank solution, standard solutions containing B or W and SiC pyrohydrolysis digests. In addition, it is possible to observe the interferences of W on the emission lines for B I 249.677 nm and B I 249.772 nm in the SiC pyrohydrolysis digest and B aqueous standard. In B I 249.677 nm, the signal for B standard is completely overlapped by the signal of W standard solution (10 mg L^{-1} of W). On the other hand, the overlap on B I 249.772 nm was not complete, but could affect the accuracy of the B determination. A tungsten signal could be observed also in SiC pyrohydrolysis digest and a concentration of tungsten in this solution was about 10 mg L^{-1}. In this sense, W has emission lines at W II 249.664 nm (difference of 13 pm in relation to 249.677 B emission line) and W II 249.748 nm (difference of 24 pm in relation to 249.772 B emission

line) that could cause interferences at B main emission lines. Significant spectral interferences of W were not observed for B I 182.639 and B I 208.889 nm emission lines. However, the relative intensity of these lines is about two times lower than the more intense emission line (B I 249.772 nm). Spectral interferences of V on B I 182.639 nm and B I 208.889 nm emission lines were also evaluated. A concentration up to 50 mg L^{-1} of V was investigated and significant interferences were not observed for the emission lines for B using ICP-OES. For B determination the emission lines at B I 182.639 and B I 208.889 nm were selected.
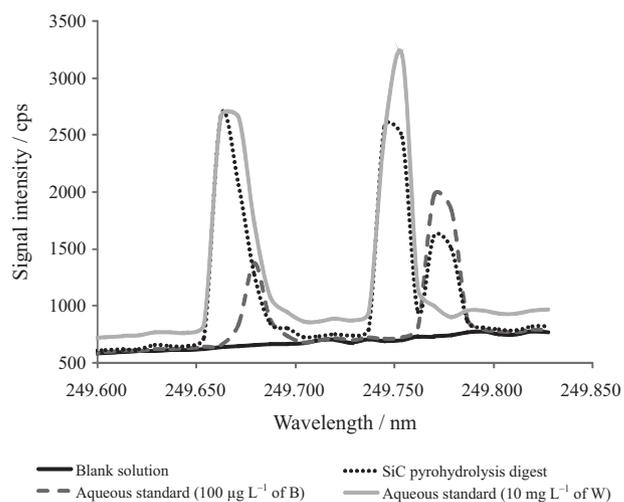

Figure 1. Spectral profiles for boron and tungsten emission lines.

Evaluation of air flow rate and water flow rate pumped through the heated alumina capillary

The air and water flow rates play an important role in the pyrohydrolysis reaction of B and the use of an appropriate air flow rate can minimize excessive cooling of the reactor tube without affecting the volatilization of analyte. On the other hand, the water flow rate should allow the generation of a suitable amount of water vapor for the complete hydrolysis of B at high temperatures. In Figures 2 and 3, results are shown for the effect of air and water flow rates for the recovery of B in the SiC sample. For air flow rate, the maximum recovery for B was observed at 0.3 L min^{-1}. Values of air flow rate higher than 0.3 L min^{-1} probably promote the cooling of the reactor and minimize the B volatilization. In addition, 1.5 mL min^{-1} of water flow rate pumped through the heated alumina capillary allows the efficient hydrolysis of B with the highest recovery for B (Figure 3). For the other values of water flow rates investigated, both an insufficient water amount for hydrolysis analysis and cooling of the reactor can explain the lowest values of B recovery. Flow rates of 0.3 L min^{-1} and 1.5 mL min^{-1} for air and water pumped

through the heated alumina capillary were selected for further B determinations, respectively.
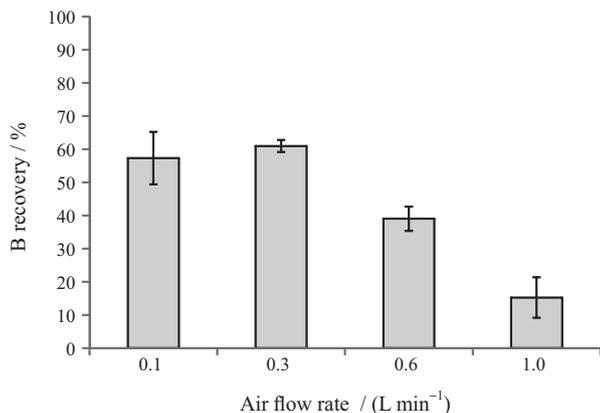

Figure 2. Evelution of air flow rate on boron recovery during pyrohydrolysis procedure. SiC mass of 200 mg, temperature of 1000 °C, water flow rate pumped through the heated alumina capillary of 1.0 mL min⁻¹ reaction time of 10 min, and without V_2O_5.

Evaluation of temperature and use of auxiliary reagent

Temperature is an important parameter that should be evaluated for the pyrohydrolysis method. In this work, temperature was evaluated ranging from 750 °C to 1100 °C. Boron recovery is dependent of temperature and to assure complete recovery of B, temperatures higher than 950 °C are necessary. Additionally, significant differences on B recoveries were not observed for temperatures of 1050 and 1100 °C. Thus, the temperature of 1050 °C was selected for further tests in order to assure maximum recovery of B and avoid that volatile species of boron remain inside the pyrohydrolysis tube after the heating cycle. Similar temperature was also used for determination of B in refractory borides[31] and alloys.[29]

One of the most used auxiliary reagents in pyrohydrolysis is V_2O_5, which aids the analyte volatilization from the matrix. In order to evaluate the effect of V_2O_5 in the proposed procedure, 200 mg of the sample was submitted to pyrohydrolysis without the use of the auxiliary reagent and increasing the amount of V_2O_5 up to 800 mg (one part of SiC + 4 parts of V_2O_5). Samples masses of V_2O_5 up to 100 mg did not improve the recovery for B. On the other hand, V_2O_5 masses equal or higher than 200 mg promote the increase of B recoveries. Significant differences (ANOVA, 95% confidence level) were not observed for B recovery using 200, 400 and 800 mg of V_2O_5. Based on these results, the use of 200 mg of V_2O_5 was selected for the pyrohydrolysis of 200 mg of SiC. Hamner and De'Aeth proposed B determination in alloys and V_2O_5 was necessary to assure the complete volatilization of B.[41] In addition, it is important to mention that, in this proposed method V_2O_5

was produced in relatively high purity from ammonium metavanadate heating and did not increase blank values.
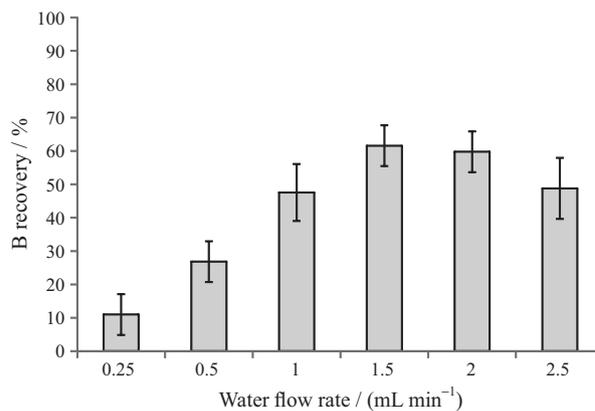

Figure 3. Evelution of water flow rate pumped through the heated alumina capillary on boron recovery during pyrohydrolysis procedure. SiC mass of 200 mg, temperature of 1000 °C, air flow rate of 0.3 L min⁻¹, reaction time of 10 min and without V_2O_5.

Evaluation of reaction time

The reaction time of pyrohydrolysis is another very important parameter and for some applications up to 2 h was necessary for complete volatilization of B.[41] In this sense, the evaluation of time ranging from 7.5 to 25 min was carried out for the SiC analysis. According to results obtained during optimization of reaction time, it is possible to observe that a minimum of 15 min is required for complete volatilization of B using the proposed pyrohydrolysis method. Thus, in order to assure that boron species were not present inside the reactor tube, a reaction time of 20 min was selected for the analysis of SiC.

Boron determination in SiC samples after pyrohydrolysis

SiC samples prepared by pyrohydrolysis were analyzed by ICP-OES and ICP-MS and the results obtained for B are shown in Table 1. Boron was only quantified in samples SiC I, SiC II and BAM S003. The B concentration was lower than LOD for samples SiC III and SiC IV. In Table 2 are provided the LODs obtained by pyrohydrolysis and microwave-assisted acid digestion with detection of B by plasma based techniques. The LODs obtained by pyrohydrolysis method were 0.25, 0.50 and 1.2 µg g⁻¹ (3s criterion) for B emission lines of B I 249.772, B I 208.889 and B I 182.639 nm, respectively (Table 2). It is important to mention that the B I 249.772 nm can be only used for SiC samples without the presence of W. On the other hand, better LOD was obtained using ICP-MS (0.010 µg g⁻¹). In addition, LODs obtained using pyrohydrolysis were lower than those obtained using microwave-assisted acid

Table 1. Concentration of B in SiC samples and CRM determined by ICP-OES and ICP-MS after microwave-assisted acid digestion and pyrohydrolysis (mean ± standard deviation, n = 3)

Sample	Microwave-assisted acid digestion			Pyrohydrolysis		
	B / (μg g^{-1}) ICP-OES 182.639 nm	B / (μg g^{-1}) ICP-OES 208.889 nm	B / (μg g^{-1}) ICP-MS ^{11}B	B / (μg g^{-1}) ICP-OES 182.639 nm	B / (μg g^{-1}) ICP-OES 208.889 nm	B / (μg g^{-1}) ICP-MS ^{11}B
SiC I	17.6 ± 1.1	18.7 ± 1.2	18.5 ± 0.9	18.0 ± 1.0	17.8 ± 0.9	18.4 ± 0.7
SiC II	15.9 ± 1.4	16.3 ± 1.3	16.0 ± 1.0	16.1 ± 0.6	16.4 ± 0.5	15.9 ± 0.4
SiC III	< 6.0	< 2.5	< 0.050	< 1.2	< 0.50	< 0.010
SiC IV	< 6.0	< 2.5	< 0.050	< 1.2	< 0.50	< 0.010
BAM S003	62.8 ± 3.0	64.0 ± 3.7	64.2 ± 2.8	62.5 ± 2.7	63.5 ± 2.0	63.2 ± 1.8

CRM BAM S003 = 63 ± 7 μg g^{-1}

digestion, because SiC wet digests containing HF that should be diluted (5 fold) in order to minimize damages on nebulization system and torch of plasma based techniques. Additionally, in Table 2 are also provided LODs reported in literature for B determination in SiC materials. Limits of detection obtained by Docekal *et al.*[8] using slurry nebulization-ICP-OES were not specified for each element, but according to the authors, LODs ranging from 0.10 to 1.0 μg g^{-1} were obtained for all analytes. On the other hand, Franek and Krivan[9] obtained LOD of 0.6 μg g^{-1} for B using microwave-assisted acid digestion and determination by ICP-OES. In addition, LOD obtained by Hamner and DeAeth,[41] for determination of B in silicon-bearing alloys and other material using pyrohydrolysis, is not specified in the manucript. In this sense, the lower concentration of B obtained for sample reported in the article was adopted as LOD (5.0 μg g^{-1}). Finally, LODs obtained using pyrohydrolysis for B determination in SiC sample were similar with those reported by other methods and suitable for quality control of SiC.

The accuracy of the proposed method was evaluated by the analysis of certified reference material (BAM S003 - silicon carbide) and bias value lower than 1% between the certified and determined values was achieved. These results show the suitability of pyrohydrolysis as sample preparation for high purity SiC samples and subsequent determination of B using ICP-OES.

Conclusions

The pyrohydrolysis method that is suitable for trace levels of B determination in SiC samples. Determination of B using pyrohydrolysis was up to 10 fold faster in comparison with conventional microwave-assisted acid digestion. Quantitative recoveries of B were obtained after 20 min of reaction time at 1050 °C and the use of deionized water to absorb the pyrohydrolysis products. The

Table 2. LODs obtained for B using pyrohydrolysis and microwave-assisted acid digestion using plasma based technique and comparison with results reported in literature

Method	LOD / (μg g^{-1})
Pyrohydrolysis using B I 249.772 nm (ICP-OES)[a]	0.25
Pyrohydrolysis using B I 208.889 nm (ICP-OES)[b]	0.50
Pyrohydrolysis using B I 182.639 nm (ICP-OES)[a]	1.2
Pyrohydrolysis using ICP-MS	0.010
Microwave-assisted acid digestion using B I 249.772 nm (ICP-OES)[a]	1.25
Microwave-assisted acid digestion using B I 208.889 nm (ICP-OES)[b]	2.5
Microwave-assisted acid digestion using B I 182.639 nm (ICP-OES)[a]	6.0
Microwave-assisted acid digestion using ^{11}B (ICP-MS)	0.050
Slurry nebulization using ICP-OES[8]	0.10 to 1.0
Wet digestion with ICP-OES[9]	0.60
Pyrohydrolysis using ICP-OES[41]	5.0[c]

[a]For samples without W; [b]for samples with W; [c]LOD of the method is not specified in article and lower concentration of B obtained in the article was used.

use of only deionized water is suitable for direct analysis by plasma based techniques minimizing problems with the nebulization system and plasma stability. Use of 200 mg of V_2O_5 mixed with the SiC was necessary in order to assure the complete volatilization of B. Boron determination using ICP-OES could be carried out. However, the main emission lines for boron can not be used due to spectral interference promoted by W. The secondary emission line at B I 182.639 and B I 208.889 nm should be used. Determination of B using ICP-MS was free from spectral interference and suitable LOD were obtained using pyrohydrolysis method. Good agreement with certified reference material was obtained and relative standard deviation was lower

than 5%. In this sense, considering the simplicity of the pyrohydrolysis apparatus and the main characteristics of the method, it can be used for purity control of SiC samples.

Acknowledgements

References

1. Neudeck, P. G.; *J. Electron. Mater.* **1995**, *24*, 283.

2. Shenai, K.; Scott, R. S.; Baliga, B. J.; *IEEE T. Electron Dev.* **1989**, *36*, 1811.

3. Trew, R. J.; Yan, J. B.; Mock, P. M.; *Proc. IEEE* **1991**, *79*, 598.

4. Demichelis, F.; Pirri, C. F.; Tresso, E.; *J. Appl. Phys.* **1992**, *72*, 1327.

5. Demichelis, F.; Pirri, C. F.; Tresso, E.; Dellamea, G.; Rigato, V.; Rava, P.; *Semicond. Sci. Tech.* **1991**, *6*, 1141.

6. Tanaka, H.; *J. Ceram. Soc. Jpn.* **2011**, *119*, 218.

7. Broekaert, J. A. C.; Brandt, R.; Leis, F.; Pilger, C.; Pollmann, D.; Tschopel, P.; Tolg, G.; *J. Anal. At. Spectrom.* **1994**, *9*, 1063.

8. Docekal, B.; Broekaert, J. A. C.; Graule, T.; Tschopel, P.; Tolg, G.; *Fresen. J. Anal. Chem.* **1992**, *342*, 113.

9. Franek, M.; Krivan, V.; *Fresen. J. Anal. Chem.* **1992**, *342*, 118.

10. Nickel, H.; Broekaert, J. A. C.; *Fresen. J. Anal. Chem.* **1999**, *363*, 145.

11. Docekal, B.; Krivan, V.; *J. Anal. At. Spectrom.* **1992**, *7*, 521.

12. Barth, P.; Krivan, V.; *J. Anal. At. Spectrom.* **1994**, *9*, 773.

13. Peng, T.; Sheng, X.; Hu, B.; Jiang, Z.; *Analyst* **2000**, *125*, 2089.

14. Schaffer, U.; Krivan, V.; *Anal. Chem.* **1999**, *71*, 849.

15. Wang, Z.; Qiu, D.; Ni, Z.; Tao, G.; Yang, P.; *Anal. Chim. Acta* **2006**, *577*, 288.

16. Broekaert, J. A. C.; Lathen, C.; Brandt, R.; Pilger, C.; Pollmann, D.; Tschopel, P.; Tolg, G.; *Fresen. J. Anal. Chem.* **1994**, *349*, 20.

17. Matschat, R.; Habler, J.; Traub, H.; Dette, A.; *Anal. Bioanal. Chem.* **2005**, *383*, 1060.

18. Botelho, G. M. A.; Curtius, A. J.; Campos, R. C.; *J. Anal. At. Spectrom.* **1994**, *9*, 1263.

19. Wei, W. C.; Chen, C. J.; Yang, M. H.; *J. Anal. At. Spectrom.* **1995**, *10*, 955.

20. Schäffer, U.; Krivan, V.; *Fresen. J. Anal. Chem.* **2001**, *371*, 859.

21. Mattos, J. C. P.; Flores, E. M. M.; Krivan, V.; *J. Anal. At. Spectrom.* **2008**, *23*, 931.

22. Byrne, J. P.; Gregoire, D. C.; Goltz, D. M.; Chakrabarti, C. L.; *Spectrochim. Acta, Part B* **1994**, *49*, 433.

23. Kiera, A. F.; Schmidt-Lehr, S.; Song, M.; Bings, N. H.; Broekaert, J. A. C.; *Spectrochim. Acta, Part B* **2008**, *63*, 287.

24. Flórián, K.; Hassler, J.; Schrön, W.; *Fresen. J. Anal. Chem.* **1996**, *355*, 601.

25. Warf, J. C.; Cline, W. D.; Tevebaugh, R. D.; *Anal. Chem.* **1954**, *26*, 342.

26. Antes, F. G.; Pereira, J. S. F.; Enders, M. S. P.; Moreira, C. M. M.; Muller, E. I.; Flores, E. M. M.; Dressler, V. L.; *Microchem. J.* **2012**, *101*, 54.

27. Morgan, L.; *Analyst* **1964**, *89*, 621.

28. Wiederkehr, V. R.; Goward, G. W.; *Anal. Chem.* **1959**, *31*, 2102.

29. Williams, J. P.; Campbell, E. E.; Magliocca, T. S.; *Anal. Chem.* **1959**, *31*, 1560.

30. McKinley, G. J.; Wendt, H. F.; *Anal. Chem.* **1965**, *37*, 947.

31. Watanabe, M.; Narukawa, A.; *Bunseki Kagaku* **2000**, *49*, 583.

32. Antes, F. G.; Santos, M. F. P.; Guimaraes, R. C. L.; Paniz, J. N. G.; Flores, E. M. M.; Dressler, V. L.; *Anal. Methods* **2011**, *3*, 288.

33. Antes, F. G.; Pereira, J. S. F.; Spadoa, L. C.; Muller, E. I.; Flores, E. M. M.; Dressler, V. L.; *J. Braz. Chem. Soc.* **2012**, *23*, 1193.

34. Dressler, V. L.; Pozebon, D.; Flores, E. L. M.; Paniz, J. N. G.; Flores, E. M. M.; *J. Braz. Chem. Soc.* **2003**, *14*, 334.

35. Langenauer, M.; Krahenbuhl, U.; Wyttenbach, A.; *Anal. Chim. Acta* **1993**, *274*, 253.

36. Ohno, T.; Muramatsu, Y.; Shikamori, Y.; Toyama, C.; Okabe, N.; Matsuzaki, H.; *J. Anal. At. Spectrom.* **2013**, *28*, 1283.

37. Schnetger, B.; Muramatsu, Y.; *Analyst* **1996**, *121*, 1627.

38. Taflik, T.; Duarte, F. A.; Flores, E. L. M.; Antes, F. G.; Paniz, J. N. G.; Flores, E. M. M.; Dressler, V. L.; *J. Braz. Chem. Soc.* **2012**, *23*, 488.

39. Fietzke, J.; Frische, M.; Hansteen, T. H.; Eisenhauer, A.; *J. Anal. At. Spectrom.* **2008**, *23*, 769.

40. Elinson, S. V.; Evseeva, T. I.; Zemlyanukhina, N. A.; Keruchenko, T. A.; Pavlova, I. V.; Chepchugova, T. Y.; Tsvetkova, V. T.; *J. Anal. Chem. USSR* **1984**, *39*, 681.

41. Hamner, R. M.; DeAeth, L. A.; *Talanta* **1980**, *27*, 535.

42. http://physics.nist.gov/asd, Kramida, A.; Ralchenko, Y.; Reader, Y.; NIST ASD Team, In NIST Atomic Spectra Database (version 5.1), Gaithersburg, MD, accessed in June 2014.

43. Rao, J. B.; Catherin, G. J.; Murthy, I. N.; Rao, D. V.; Raju, B. N.; *Int. J. Eng. Sci. Technol.* **2011**, *3*, 82.

44. Belton, G. R.; McCarron, R. L.; *J. Phys. Chem.* **1964**, *68*, 1852.