The precise determination of nitrogen in boron nitride

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Abstract

In order to further improve the high performance ceramic material BN it is necessary to advance its analytics. The quantitative determination of the nitrogen content as main component is compared by three methods: the carrier gas hot extraction, the LiOH fusion and the Kjeldahl method. Thereby specific methodical aspects are critically highlighted with respect to the trueness and precision of the nitrogen analysis. The "chemistry" plays a fundamental role in all methods. In the case of the instrumental analysis by CGHE important critical aspects are the calibration of CGHE measurements and the improvement of reproducibility which needs a much better understanding of the chemical reactions in the crucible. In case of the Kjeldahl method it is of decisive importance to apply an adequate high temperature of 260 °C for decomposition, whereas the melt-decomposition with LiOH is affected by melt additions.
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1. Introduction

Boron nitride is an important advanced ceramic material. There are two major allotropes analogous to those of carbon. α-BN has a hexagonal structure similar to graphite and β-BN has a cubic structure similar to diamond. For the preparations of the most widely used α-BN (further designated as BN) on industrial scale, reactions of boron oxide or boric acid with ammonia or with organic nitrogen compounds (e.g., urea or melamine) are used. BN has a unique combination of advantageous properties like high temperature stability (melting point: 2730 °C), chemical inertness against acids and molten metals, low density, excellent lubrication properties and stability to thermal shock. It is stable in air up to 1000 °C and a very good electric insulator. As a thermal conductor BN is about as efficient as stainless steel. Due to its non-wetting properties it can be used as a container material for molten silicon, boron, glass and salts and reactive metals. On top of this hot-pressed shapes are easy to machine with conventional tools.[1,2] Important applications of hot-pressed BN shapes are insulators for high temperature furnaces, break rings and side-dams for casting of steel and alloys, moulds for alloys, tools and parts for the glass industry and crucibles. It is also used for industrial spray coatings and as active filler for cosmetics.[3]

In order to further improve this high performance ceramic material it is necessary to advance the analytics of BN. In this field not only the determination of trace impurities is important but also the precise determination of main components.[4] In this paper we compare the results of the quantitative determination of the nitrogen content in a commercial BN powder by three methods: the widely used carrier gas hot extraction method (CGHE), the LiOH fusion and the Kjeldahl method. Thereby specific methodical aspects are critically highlighted with respect to the trueness and precision of the nitrogen analysis as a main component.

- The application of the CGHE (or inert gas fusion analysis) method with respect to the nitrogen determination is based on the thermal decomposition of the nitride sample, mostly in the presence of a melt forming metal addition:

$$BN \rightarrow [B]_{melt} + 1/2 N_2 \tag{1}$$

This simplistic reaction type, however, is depending on a lot of experimental parameters. Especially in the case of the

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high temperature stable BN, the process is influenced by those experimental conditions as maximum heat input by electrical heating of a graphite crucible reactor, which is defined by furnace and graphite crucible types used, as the realisation of metal additions, which fulfil a function as capsule material necessary for powder containment and as melt former.[5] After the complete formation of the gaseous reaction product the nitrogen has to be extracted from the liquid melt into the gas phase and transferred by the helium carrier gas to a thermal-conductivity detection cell. The determination of the oxygen concentration as impurity is usually carried out simultaneously and based on the reaction of the oxygen sources of the BN sample (oxide impurities, surface effects by weathering) with the carbon of the graphite crucible, enhanced by melt conditions and forming either CO_2 or CO. Their detection takes place selectively by non-dispersive infrared cells.

- Sample decomposition by melt-fusion with alkali-hydroxides is a very old analytical technique which was first applied by the German chemist Martin Heinrich Klaproth in 1790.[6] Compared to the similar melt-fusion with alkali-carbonates, melt-fusion with alkali-hydroxides can be performed at significant lower temperatures. Inorganic nitrides decompose in the alkali-hydroxide melt under formation of ammonia which is transported by means of a stream of inert gas to an absorption vessel containing boric acid solution:

$$BN + 3LiOH \rightarrow Li_3BO_3 + NH_3 \tag{2}$$

The amount of ammonia in the sample solution is determined by titration with an acid of known concentration. The method needs no calibration and provides excellent accuracy and precision. Although this procedure is very time consuming it has to be considered as a reference method to check the accuracy of modern analytical methods like CGHE. For the determination of total nitrogen in BN lithium-hydroxide (LiOH)[7] and sodium-hydroxide[8] were used. Because of better handling properties LiOH was used in this work (LiOH can be purchased as fine, non-hygroscopic powder).

- The Kjeldahl method was developed 1883 by the Danish chemist Johan Kjeldahl.[9] Originally used for the nitrogen determination in animal and plant samples the method soon was used for inorganic substances too. Using Kjeldahl method nitrogen is transformed in ammonia by a digestion and driven out of the solution by concentrated sodium hydroxide solution. In case of BN the reaction path for decomposition is as follows:

$$BN + 4HF \rightarrow [NH_4]_{solv}{}^+ [BF_4]_{solv}{}^- \tag{3}$$

As can be seen in Section 3, complete decomposition is decisive for correct values but not easy to reach. The generated ammonia is then distilled using a steam distillation into a flask containing a surplus of sulphuric acid. The surplus of acid is then titrated with sodium hydroxide solution to determine the amount of nitrogen in the sample precisely. Though the method is quite old, the results are of high quality as far as precision is concerned.

In this paper the results of an interlaboratory study on the determination of nitrogen and oxygen in high-purity BN powder are presented. The study was carried out in the working group "Sonderwerkstoffe" (special materials) of the Chemist's Committee of the Gesellschaft für Bergbau, Metallurgie, Rohstoff- und Umwelttechnik (GDMB).

2. Experimental

2.1. Boron nitride sample

The BN sample 94385 is a high-purity powder produced and characterised by H. C. Starck GmbH Laufenburg, Germany. Its content of soluble boric oxide is below 0.1%. The oxygen content is about 0.7%. The relatively fine powder has an average particle size of about 0.3 μm and a specific surface area of 4.0 m²/g. The apparent density of the powder is only 166 g/l.

2.2. Carrier gas hot extraction method

The CGHE analysis was carried out with commercial simultaneous oxygen/nitrogen analysers of various types and producers (TC 436, TC 436DR, TC 436Ar, TCH 600 from LECO, USA and ONH 2000 from ELTRA, Germany). Typical parameters are listed in Table 1. The BN powders were weighed in a Sn capsule (mostly) or a Ni capsule on a semi-microbalance. Together with Ni as crucible (both capsules are then pressed together forming a "sandwich") or as separate addition (basket, pin) the sample was analysed. Following an out-gas cycle, the crucible temperature was lowered by reducing the heating power or current and the sample was transferred to the hot crucible by a sample drop mechanism. Mostly, the temperature was increased power or current controlled by the ramping mode with a given ramping rate. In some cases the so-called impulse mode was used where the sample drops in the crucible held at a selected reaction temperature. There is only an inexact power/current–temperature correlation depending on the instrumental state, the quality of the graphite crucible and its geometry etc. The actual situation to reach the highest possible temperature of about 2800 °C by the applied instrument has to be checked. The whole analytical process (reaction-detection) has to be calibrated. Unfortunately, no reference materials of the same matrix exist. Thus, the fundamental part of the reaction itself is not proved when using reference materials with different matrix composition such as Si_3N_4 (S001, BAM Berlin) which has been used for TC-detector calibration.

2.3. Melt-fusion method

The sample decomposition was performed with an IRF-10 temperature programmable infrared tubular furnace (Fa. Behr, Germany). Unglazed porcelain boats in the dimension 90 mm × 12 mm × 10 mm with lid were used for melt-fusion (Fa. Haldenwanger, Germany). The determination of ammonia in the sample solutions was performed with a potentiometric titration system consisting of Titrando 809, Dosino 800 and Profitrode pH-electrode (Metrohm, Germany). The tem-

Table 1
Experimental conditions of the CGHE method.

Lab	Analyser type	Crucible type	Sample mass [mg]	Sn additive[a] [g]	Ni additive [g]	Heating parameters	Ramp [W/s]
1	TC436	782-720[b]	15	0.16	0.5[c]	3000–5000 W	30
2	ONH 2000	CR-520[d]	1–1.5	0.017	1[e]	4500 W	no[f]
3	TCH 600	782-720[b]	10–30	–	0.23[a]	5000 W	no[f]
4	TC 436DR	782-720[b]	20	0.16	0.4[a]	1000–5000W	50
4	TC 436DR	782-720[b]	20	0.16	0.4[a]	4000–5500 W	30
5	TC 436DR	776-247[d]	13	0.25	0.5 + 0.1C[g]	1075 A	no[f]
6	TC 436DR	782-720[b]	0.85	–	0.070[a]	3000–5700 W	11
7	TC 436Ar	782-720[b]	10	0.016	1[e]	4500–6000 W	30
8	TC 436	782-720[b]	15	0.16	1[e]	900–950 A	10[h]
8	TCH 600	782-720[b]	20–30	0.16	–	4000–5200 W	100

[a] Closed capsule.
[b] High temperature crucible.
[c] Pin.
[d] Standard crucible.
[e] Basket.
[f] no = IMPULSE heating.
[g] 0.5 g Ni-pin + addition of 0.1 g carbon powder.
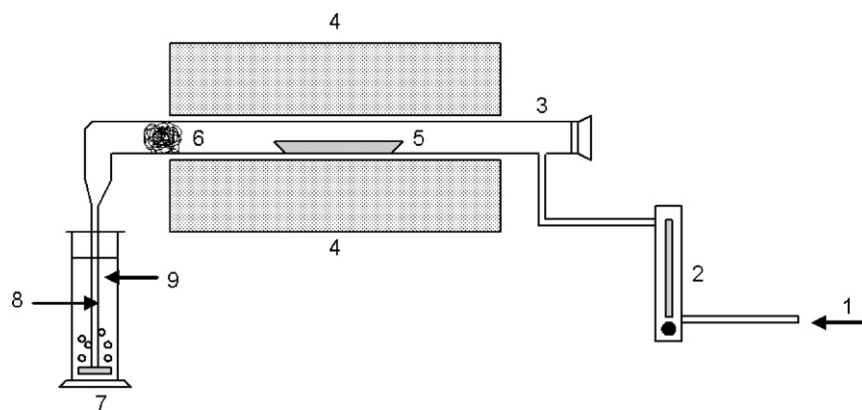[h] RAMP in [A/s].

Fig. 1. Digestion apparatus for melt-fusion: (1) inert gas; (2) flow meter (0–200 ml/min); (3) vitreous silica reaction tube with connections; (4) IRF-10 tubular furnace; (5) porcelain boat with sample and LiOH; (6) vitreous silica wool; (7) absorption vessel (150 ml); (8) gas inlet tube with frit; (9) boric acid solution.

perature/time program applied for sample digestion is listed in Table 2. The set-up of the digestion apparatus is shown in Fig. 1. The following reagents were used:

- powdered lithium hydroxide, LiOH (Merck, Germany)
- titration solution: hydrochloric acid, HCl, 0.1 m (Merck, Germany)
- boric acid, H_3BO_3 (Merck, Germany)
- sodium carbonate, Na_2CO_3 (Merck, Germany)
- inert gas, nitrogen, purity $\geq$ 99.99% (Fa. SWF, Germany).

Boric acid solution was prepared by dissolving 40 g of H_3BO_3 in 1 l of hot water.

Table 2
Temperature/time program for sample digestion via melt-fusion.

Step	Ramp [min]	Hold [min]	Temperature [°C]
1	10	5	450
2	10	0	650
3	0	30	650

Before analysis, the BN sample was dried at $120\,°C \pm 5\,°C$ for a minimum of 2 h. After cooling, the sample was stored in a desiccator. All reagents were of known analytical grade and it was ascertained that the reagents were of sufficiently high purity to permit their use without lessening the accuracy of the determination. Distilled water was used throughout.

The porcelain boats were coated by weighing 0.5 g of LiOH into the boats followed by heating in a muffle furnace for 4 h at 650 °C. After cooling, the coated boats were stored in a desiccator. App. 45 mg of BN was weighed into a coated boat and 0.1 g of boric acid was added. BN and boric acid were mixed thoroughly using a micro-spatula. After that, 0.5 g of LiOH was added and again mixed. The mixture was then covered with 0.5 g of LiOH. The porcelain boat with the reaction mixture was covered with a porcelain lid, inserted in the reaction tube and placed in the middle of the heating zone of the furnace. The reaction tube was closed and the inert gas flow was set to a rate of 80 ml/min. The absorption vessel was filled with 40 ml of boric acid solution and 60 ml of water, the gas inlet tube immersed and connected to the outlet of the reaction tube and the temperature/time pro-

gram of the furnace was started. After sample decomposition, the gas inlet tube was disconnected and thoroughly rinsed inside and outside with water. The sample solution in the absorption vessel was quantitatively transferred into a glass beaker. The amount of ammonia in the samples solution was determined by titration to the equivalence point with 0.1 m HCl using the potentiometric titration system. The equivalence point was generally a pH value of 4.6 ± 0.2. The titration correction factor of 0.1 m HCl was determined using sodium carbonate as primary substance (dried at $120\,^{\circ}$C for 2 h, sample mass app. 150 mg). According to the described procedure also the method-blank was determined. The accuracy was checked using a silicon nitride certified reference material, ERM® ED-101 (BAM, Germany). The nitrogen content was calculated as wt.% using the following Eq. (4):

$$N_{total}\ (\%) = \frac{V_{acid}\,Ff \times 100}{m_S} \tag{4}$$

V_{acid} = consumption of 0.1 m HCl [ml]; F = 1.4007 (titrimetric factor in mg N/ml 0.1 m acid); f = titration correction factor of acid; m_{sample} = sample mass [mg].

2.4. Kjeldahl method

About 0.2 g of the BN sample was weighed in a PTFE-container with 10 ml of HF-acid (40%). This sample was decomposed for 20 h at a temperature of $260\,^{\circ}$C in a high pressure decomposition device (Berghof Products and Instruments GmbH, Germany). After completion of the decomposition the cold solution was filled into a volumetric flask (100 ml). About 20–30 ml of the solution was weighed (m_s) in a sample cup and transferred into the Kjeldahl container. This container was built into the distillation device of model Vapodest 3 (Gerhardt Laboratory Systems, Germany). About 30 ml sulphuric acid (0.05 mol/l) was titrated into an Erlenmeyer flask and diluted with approximately 120 ml of water. Then, 2–3 ml of indicator solution (0.2% methyl red + 0.1% methylene blue in ethanol p.a.) was added. Then the solution of sodium hydroxide (40%) was applied to the sample and distilled with steam into the Erlenmeyer flask. After completion of the distillation the excessive sulphuric acid was titrated with sodium hydroxide solution (0.1 mol/l):

$$N_{total}\% = \frac{(V_{H_2SO_4} f_{H_2SO_4} - V_{NaOH} f_{NaOH})F}{m_S} \tag{5}$$

V_{acid} = consumption of 0.1 m H_2SO_4 [ml]; F = 70,945 (titrimetric factor with 0.2 g solid sample); $f_{H_2SO_4}$ = titration correction factor of $H_2 2SO_4$ solution; f_{NaOH} = titration correction factor of NaOH solution; m_s = sample mass of taken liquid [mg].

Precise value of the molarity of the sulphuric acid was determined titrating sodium carbonate primary substance. The molarity of the sodium hydroxide was determined with this sulphuric acid. This way there is no doubt about the accuracy of titration.

3. Results and discussion

3.1. Carrier gas hot extraction

Because the nitrogen analysis is typically combined with the oxygen determination, both analytical signals from the TC and the IR detectors were under examination to get information about the progress of the reaction. Applying a short ramping mode, e.g., starting from already high temperatures (e.g., about $2400\,^{\circ}$C as corresponding to 4000 W) with 30 W/s to 5500–6000 W, the sample reacts very fast. As seen in Fig. 2, the conversion of oxygen species in BN creates shortly after sample drop an O signal in a complex melting – carbothermal reduction – CO formation process. The generation of nitrogen is dragged on time and higher temperatures. The shown detector curves are delay time corrected in comparison to the power–time relation.

The volume of nitrogen gas released from the BN of about 15 mg sample weight corresponds to about 10 ml at room temperature and normal pressure. At the high temperatures in the reaction crucible this volume expands dramatically. Depending on the individual instrumental design as tube diameters, flow regulation etc. a pressure impulse may influences the gas flow in the system. Because the detectors are sensitive to flow irregularities with respect to the signal – concentration detection function, the fast gas formation may cause erroneous analytical results with respect to trueness as reproducibility. The same critical remarks have to be made about the so-called impulse mode for nitrogen analysis in nitrides where the sample drops into the graphite crucible held on the high reaction temperatures. Therefore, two alternatives exist: firstly, the decrease of BN amount and, secondly, the retardation of nitride decomposition by lower starting temperatures. Nowadays the weighing of small sample masses with high precision is not a problem with modern balances. But with lowering the sample aliquot the homogeneity of materials and the representation become more important. The alternative, a long ramping mode from 1000 to 5000 W within 50 W/s results in detection curves is shown in Fig. 3.

The gas formation of both CO and N_2 is broadened over time and, very interesting details of reaction kinetics become visible. At the O signal of Ni(Sn) blank material two processes are
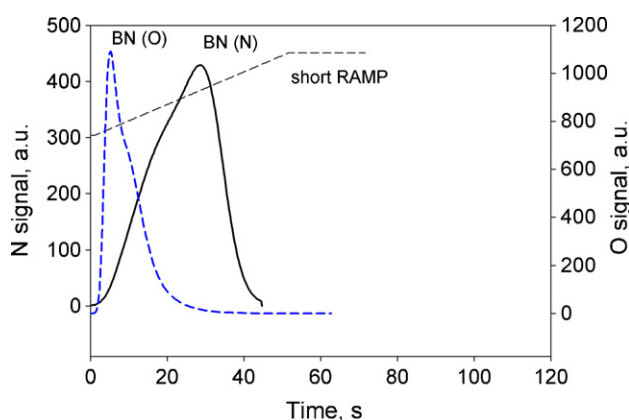

Fig. 2. Detection curves of the TC and CO_2 detectors during the BN reaction at the short ramping mode (4000–5500 W, 30 W/s).
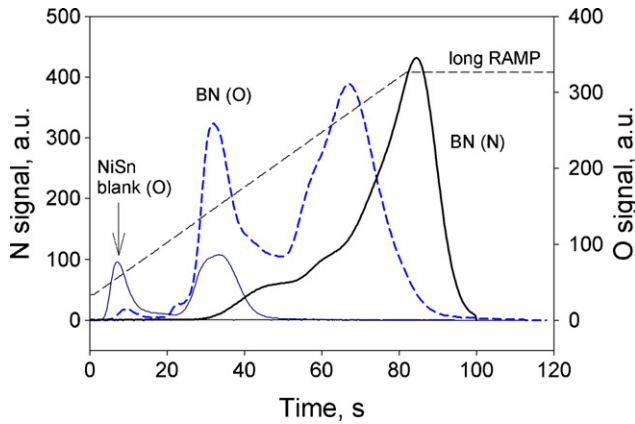
Fig. 3. Detection curves of the TC and CO$_2$ detectors during the BN reaction at the long ramping mode (1000–5000 W, 50 W/s).

detectable whereas the second one corresponds to the Ni melting. The nitrogen release from BN already starts at astonishing "low" temperatures in the nickel–tin melt. The shoulder in the N signal curve at the short ramping mode (Fig. 3) can be interpreted on this note. The nitrogen release is completed at high temperatures. Simultaneously, the formation of oxygen reaction product CO is clearly split into two parts. Its interpretation as different oxygen species in/at the BN powder is difficult: boron oxide adherently (about 0.1 wt.%) on the surface and/or 'lattice dissolved' oxygen?

A basic rule in analytical chemistry, the calibration of the whole analytical process with a reference material with the composition and concentration just like the unknown sample, is impossible to realize in the present case with solid reference materials. Therefore, the calibration of the instruments with respect to nitrogen was carried out with a certified Si$_3$N$_4$ powder material. The reaction curves of Si$_3$N$_4$ in the long ramping mode are demonstrated in Fig. 4. There is fast reaction kinetics of surface oxide reduction and complete nitride decomposition at lower temperatures than for BN, as expected. Two often discussed aspects in the CGHE method have to be pointed out: the calibration with the same amount of analyte (e.g., nitrogen) or with the same detector signal (height) of the
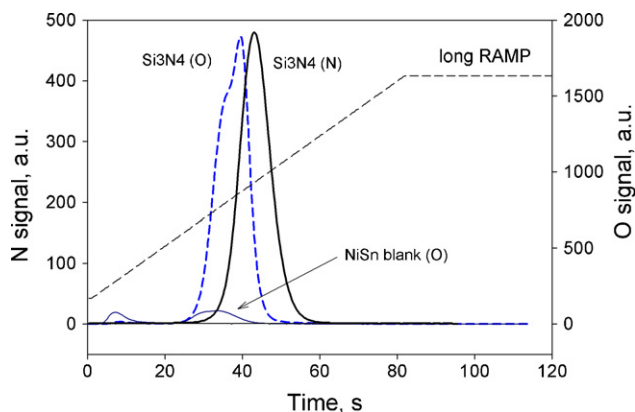

Fig. 4. Detection curves of the TC and CO$_2$ detectors during the Si$_3$N$_4$ reaction at the long ramping mode (1000–5000 W, 50 W/s).
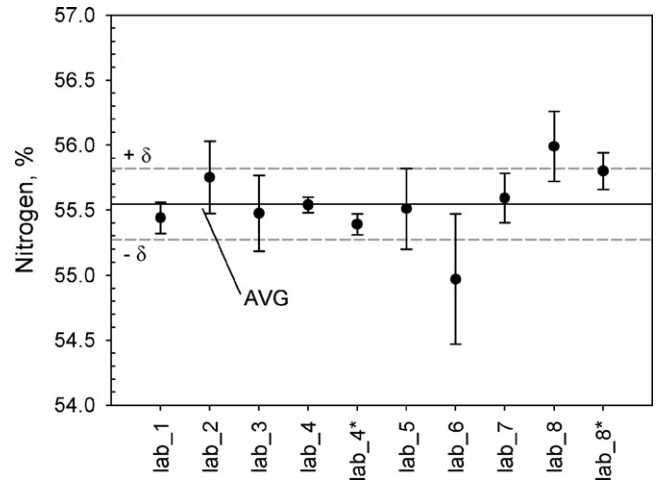

Fig. 5. Interlaboratory results of nitrogen determination of the BN 94385 sample.

TC cell? The mass dependence of signal heights is nearly linear. At a BN sample mass of 15 mg the corresponding amount of Si$_3$N$_4$ with respect to the same nitrogen amount is about 22 mg. The maximum N signal heights of the BN reaction under both ramping conditions do not differ significantly because of the self-limited thermal decomposition of BN with about 400–500 detection units. The similar signal height is reached with 10 mg Si$_3$N$_4$ compared to 750–800 detection units with 22 mg Si$_3$N$_4$. As proved statistically the calibration factor for both amounts differs at a given analyser, 1.868 ± 0.008 and 1.854 ± 0.005, respectively. This difference effectuates the analytical result of 55.6% N and 55.2% N, respectively, assuming the same measurement value of BN. For the calibration of oxygen values commercial solid reference materials with steel matrix were used.

The quantitative results of the investigations of the nitrogen and oxygen determination in BN are summarized in Table 3 and illustrated in Figs. 5 and 6. Some variations of experimental parameters with respect to the above discussion have to be stated (see Table 1):
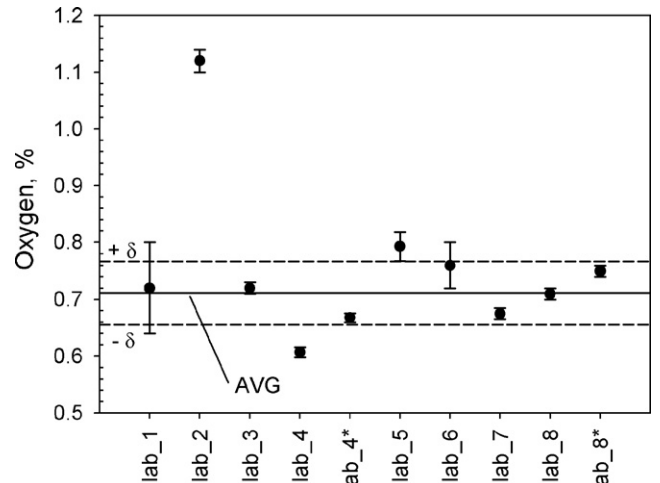

Fig. 6. Interlaboratory results of oxygen determination of the BN 94385 sample.

Table 3
Results of the nitrogen and oxygen determination in BN 94385 using the CGHE method.

Lab	Analyser type	Sample mass [mg]	N_AVG [wt.%]	N_S.D. [wt.%]	Calibration	O_AVG [wt.%]	O_S.D. [wt.%]
1	TC436	15	55.44	0.12	21[a]	0.72	0.08
2	ONH 2000	10–20	55.75	0.28	Gas dose	1.12	0.02
3	TCH 600	15	55.48	0.29	10-30[a]	0.72	0.01
4	TC 436DR	15	55.54	0.06	15[a] (vs. peak_TC)	0.61	0.01
4	TC 436DR	15	55.39	0.08	22[a] (vs. mass_N)	0.67	0.01
5	TC 436DR	12	55.51	0.31	30[a]	0.79	0.02
6	TC 436DR	0.85	54.97	0.50	3.3 mg KNO_3	0.76	0.04
7	TC 436Ar	10	55.59	0.19	20[a]	0.68	0.01
8	TC436	15	55.99	0.27	22[a]	0.71	0.01
8	TCH 600	20–30	55.80	0.14	20–50[a]	0.75	0.01

AVG – average, S.D. – standard deviation.

[a] mg Si_3N_4 certified reference material (ERM®-ED101).

- The minor reproducibility in nitrogen determination may be produced by the very small sample weight (homogeneity?) in combination with a slow ramping rate (lab_6) or by the impulse mode (lab_2, _3, _5 and lab_8 (TC 436)).
- The strong deviation of oxygen determination by lab_2 may result from non-optimised parameters using an analyser from another producer.
- Untypical conditions did not produced false values, as demonstrated by lab_8 (TCH 600) with no addition of bath material (besides the Sn capsule) and by lab_5 with lower energy insertion by using the normal crucible type instead of the usually used high temperature crucibles. The interpretation is that the carbide formation facilitates the BN decomposition due to the direct contact of BN powder with the graphite crucible wall or the addition of excess carbon powder.

3.2. Melt-fusion method

Although the melt-fusion method is not very difficult to carry out some aspects have to be considered which may influence the accuracy of the results. To condensate any lithium hydroxide which evaporates, the reaction tube outside the tubular furnace and adjacent to the absorption vessel has to be packed with loose vitreous silica wool. The silica wool is placed at a still hot part of the reaction tube. This prevents condensation of water and, thus, absorption of ammonia. Spattering of small drops of the LiOH melt can be avoided by covering the porcelain boat with a lid and by setting the heating phase up to the decomposition temperature to a minimum of 20 min. The inherent non-wetting property of BN against melts seems to be counterproductive to the application of the melt-fusion method.

In practice, the LiOH melt obviously gets in contact with every BN particle resulting in a complete decomposition of the sample. However, for some BN materials systematic lower nitrogen contents were obtained. A possible explanation could be that larger BN agglomerates were not completely decomposed. In general, an additive which improves the wettability of BN should be beneficial for a complete decomposition of the BN sample. In our experiments, the decomposition was carried out without and with addition of boric acid. The effect of boric acid can be seen in Fig. 7 and Table 4. Without addition of boric acid the residues of the decomposed BN sample can be clearly distinguished from the molten LiOH and the solidified melt looks very inhomogeneous. The low nitrogen content indicates incomplete sample decomposition. The addition of boric acid obviously improves the wetting behaviour of BN resulting in a homogeneous melt, complete sample decomposition and, thus, significantly higher nitrogen content. Table 4 also contains the results obtained for the certified reference material. The excellent agreement with the certified nitrogen content confirms the high accuracy and precision of the melt-fusion method.

Fig. 7. Solidified LiOH melts after sample decomposition. (A) Without addition of 0.1 g of boric acid; (B) with addition of 0.1 g of boric acid.

Table 4
Nitrogen concentration [wt.%] determined by lab_8 in BN 94385 and silicon nitride ERM®-ED101 (certified nitrogen concentration: 38.1 ± 0.2 wt.%) by melt-fusion method.

Decomposition no.	N (BN 94385)		N (ERM®-ED101)
	Without H_3BO_3	With H_3BO_3	with H_3BO_3
1	54.67	55.65	38.13
2	54.23	55.61	38.19
3	55.02	55.70	38.21
4	54.97	55.70	38.18
5	55.25	55.81	
6	54.66	55.70	
7	54.31	55.74	
8	55.17	55.73	
Mean value	54.79	55.71	38.18
Standard deviation	0.38	0.06	0.03
R.S.D. [%]	0.70	0.11	0.09

Table 5
Nitrogen concentration [wt.%] determined by lab_1 and lab_6 in BN 94385 by Kjeldahl method after digestion with different decomposition temperatures.

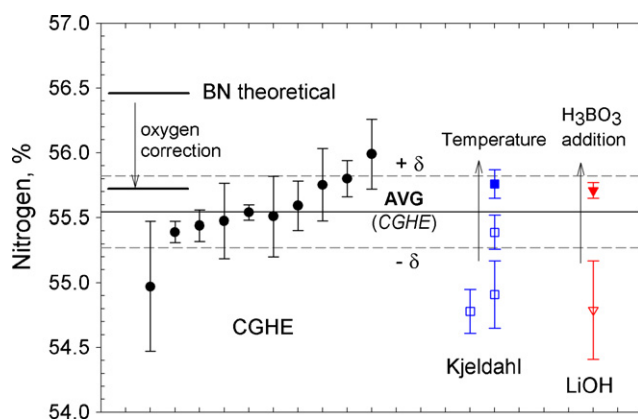| Decomposition parameters	lab_1 200 °C/20 h	lab_5 200 °C/12 h	lab_5 240 °C/12 h	lab_5 260 °C/12 h
	54.58	55.14	55.37	55.95
	54.72	54.84	55.36	55.76
	54.86	54.62	55.54	55.68
	54.96	54.75	55.40	55.74
		55.22	55.25	55.79
			55.20	55.65
			55.39	
			55.59	
Mean value	54.78	54.91	55.39	55.76
Standard deviation	0.17	0.26	0.13	0.11
R.S.D. [%]	0.31	0.47	0.23	0.20

Fig. 8. Comparison of the nitrogen content determined by different methods.

3.3. Kjeldahl method

In the first round of the round robin test a decomposition temperature of 200 °C was used. Two laboratories used the Kjeldahl-method and resulted with comparable values. From plausibility point of view and in comparison with alternative methods the results seemed too low yet. For this reason a series of determinations were accomplished using higher decomposition temperatures of 240 °C and even 260 °C. Significantly higher mean values were determined (Table 5).

4. Conclusion

As a result of the interlaboratory study it can be recognized, that the nitrogen content in high-purity α-BN-powder can be determined with all three applied methods (Fig. 8). In the case of the instrumental analysis by CGHE the variation of values seems to be larger in comparison to the other methods. But this situation may be attributed to the fact that much more laboratories took part using different specific parameters which were not optimised in relation to each other. Important critical aspects are the calibration of CGHE measurements and the improvement of reproducibility which needs a much better understanding

of the chemical reactions in the crucible. The "chemistry" plays a fundamental role in the classical methods too. In case of the Kjeldahl method it is of decisive importance to apply an adequate high temperature of 260 °C for decomposition, whereas the melt-decomposition with LiOH is affected by melt additions.

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