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Thermal decomposition of B–N–H compounds investigated by using combined thermoanalytical methods

F. Baitalow[a,*], J. Baumann[a], G. Wolf[a], K. Jaenicke-Rößler[b], G. Leitner[b]

[a]*Department of Physical Chemistry, Freiberg University of Mining and Technology, Leipziger Str. 29, D-09596 Freiberg, Germany*
[b]*Fraunhofer Institute of Ceramic Technologies and Sintered Materials Dresden, Winterbergstr. 28, D-01277 Dresden, Germany*

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Abstract

The thermal decomposition of borazane BH_3NH_3 in the temperature range up to 500 K has been studied by differential scanning calorimetry (DSC) and thermogravimetry (TG) combined with the FTIR spectroscopic and mass spectrometric analysis of the gas phase. Above 340 K borazane is decomposed in stages as the temperature is increased. The exothermic decomposition is accompanied by the release of approximately 2.2 mol H_2/mol BH_3NH_3. This corresponds to a remarkable hydrogen storage density of 14.3 mass% related to the mass of borazane. In the gas phase above the solid decomposition residue monomeric aminoborane (BH_2NH_2), borazine $(BHNH)_3$, which is the boron–nitrogen analog of benzene, and traces of diborane B_2H_6 were found beside hydrogen. The release of significant quantities of borazine was observed only at temperatures above 400 K.

Keywords: Borazane; Aminoborane; Borazine; Thermal decomposition; Coupled TG/MS

1. Introduction

Ammonia-borane (BH_3NH_3), which is also known as borazane, is a stable solid with high hydrogen content (about 20 mass%). It has been shown [1] that a thermally activated decomposition of borazane takes place in the temperature range 350–410 K accompanied by hydrogen release and heat evolution. Moderate decomposition temperatures and the exothermic character of the decomposition process allow interesting applications of borazane as a hydrogen source for fuel cells.

Such applications require reliable information about the decomposition mechanism. The thermal decomposition of borazane proceeds in two partially overlapping steps [2]. These steps can be time-resolved, if experiments are performed at low heating rates (far below 1 K min^{-1}) or at stepwise rising temperatures. The first decomposition step was separated in this way from the second step and has been characterized using different analyzing techniques [3]. It was found that borazane completely decomposes below an apparent melting point given earlier [1,4] at 385 K. It seems probable that several reactions take place simultaneously to give different solid and gaseous decomposition products [3].

In this work the thermal decomposition of borazane in the temperature range up to 500 K was studied using differential scanning calorimetry (DSC), thermogravimetry (TG), volumetric measurements, combined

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[*] Corresponding author. Tel.: +49-3731-39-2137; fax: +49-3731-39-3588.
E-mail address: felix.baitalow@chemie.tu-freiberg.de (F. Baitalow).

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TG/MS and TG/FTIR techniques. Gaseous reaction products were detected and were assigned to obtained decomposition steps. The aim of the investigations was the better understanding of the mechanism of the thermal decomposition of borazane.

In addition, information about gaseous decomposition products is of great importance for the consideration of the mechanism of the chemical vapor deposition (CVD) of cubic boron nitride (c-BN). Borazane (BH_3NH_3) and similar boron–nitrogen compounds were used as single-source precursors for the deposition of c-BN in plasma [5–9]. The mechanism of the plasma-assisted CVD of c-BN is unclear so far. It has been assumed [10] that the formation of metastable BNH_x radicals in the hydrogen plasma plays an important role in the nucleation and growth of c-BN. We want to investigate whether such reactive species also occur during the thermal decomposition of borazane.

2. Experimental

The thermal decomposition of BH_3NH_3 was monitored using a differential scanning calorimeter DSC C-80 from Setaram in the non-isothermal and isothermal mode. Experiments were performed under nitrogen atmosphere with about 20 mg borazane. Steel cells were used, which are connected to a volumetric equipment outside the calorimeter via a gas transfer line. In this way the volume of released gases was monitored simultaneously to the detected heat flow. The gas volume was measured in a calibrated all-glass apparatus by water displacement at atmospheric pressure and 298 K. Water-soluble gaseous products and gases, which condense at room temperature, were not detected by this equipment. By means of a TG–DSC 111 device from Setaram the mass loss of the solid and the heat flow were detected simultaneously. Experiments were performed under argon gas flow (flow rate 20 ml min^{-1}), the initial borazane mass was typically about 5 mg.

Combined TG/FTIR technique was applied to analyze the gaseous decomposition products. The mass loss of the solid was monitored using a thermal analyzer TGA/SDTA 851e (Mettler-Toledo) under argon flow (30 ml min^{-1}). The qualitative online FTIR analysis of the released vapors was performed using Protege 460 (Nicolet Instrument). Spectral regions for chemigrams are appointed using single spectra evaluations and comparisons to known spectra of pure gaseous components. Spectral data were collected with 16 scans/min and a resolution of 4 cm^{-1}.

Mass spectrometry was used as a complementing thermoanalytical method. The gas-phase composition was monitored using a thermal analyzer Netzsch STA 409 equipped with a quadruple mass spectrometer Balzers QMG 420 via a two-stage pressure reduction system with a stainless steel skimmer. This equipment allows the detection of unstable products immediately after their formation (within subseconds). The system was evacuated and flooded with high purity argon for three times. Then measurements were done under argon (83 ml min^{-1}) in the non-isothermal and isothermal mode. Both TG/DTA signals and the signals of the mass spectrometer in SCAN and SIM (selective ion monitoring) mode were recorded.

Borazane (BH_3NH_3) was prepared from ammonium carbonate and sodium boronhydride in a tetrahydrofuran solution according to a method described elsewhere [4]. The purity of the borazane sample was verified by powder X-ray diffraction analysis and elemental analysis.

3. Results and discussion

3.1. DSC

Calorimetric measurements of the thermal decomposition of BH_3NH_3 at temperatures up to 473 K with heating rates selected in the range of 1–10 K min^{-1} show two significant exothermic events referred to two decomposition steps (Fig. 1). The first exothermic process starts below 370 K and reaches a maximum in the temperature range 380–390 K. A partial compensation by an endothermic process is observed near 375 K. As mentioned earlier [3], this endothermic event can be assigned to the melting of borazane. Just before the first exothermic event is completely finished a further exothermic process begins. It refers to the second step of the thermal decomposition of borazane and proceeds in the temperature range up to 450 K. Separation of both decomposition steps is not attained owing to the low reaction rate.

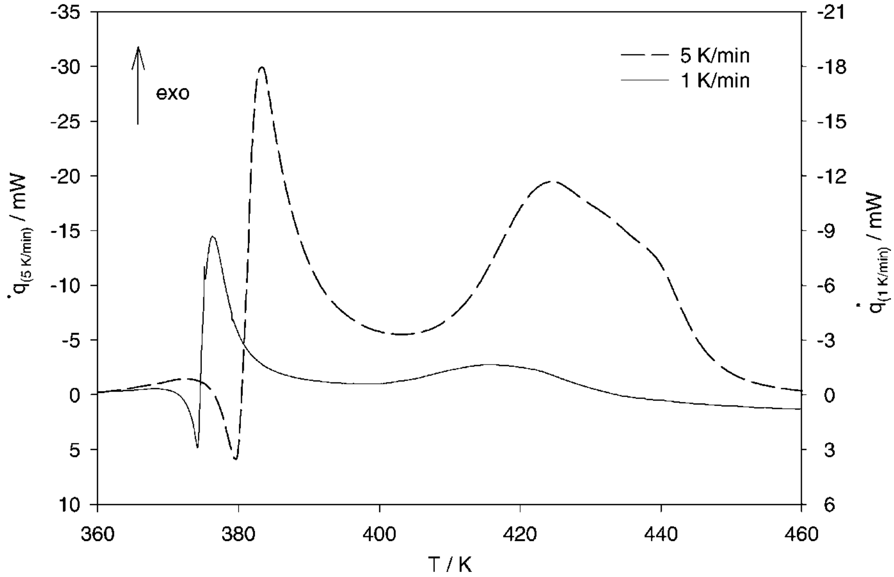

Fig. 1. Temperature dependence of the heat flow for the thermal decomposition of BH_3NH_3 (Setaram DSC 111, argon flow, heating rate 1 K min^{-1} (—), 5 K min^{-1} (- - -)).

The first decomposition step was separately observed under isothermal conditions at temperatures selected in the range of 343–363 K. For all temperatures a single exothermic process was detected in agreement with results of earlier investigations [3]. After the termination of the exothermic process (the reaction time at 363 K is more than 10 h) borazane is completely decomposed as confirmed by means of X-ray diffraction analysis. The solid decomposition residue, which mainly consists of polymeric aminoborane $(BH_2NH_2)_x$, was then heated up to 473 K using heating rates selected in the range of 0.1–1 K min^{-1}. For all heating rates we have obtained a single exothermic process referred to the second decomposition step. A typical DSC plot with an isothermal part at 363 K, followed by a non-isothermal part with heating rate 0.1 K min^{-1}, is shown in Fig. 2. Therefore, the initial decomposition of borazane under isothermal conditions allows the clear separation of both decomposition steps.

3.2. Volumetric and thermogravimetric analysis

Both exothermic steps of the thermal decomposition of borazane are accompanied by hydrogen release [2,3]. Volumetric measurements are suitable for the monitoring of the released amount of hydrogen according to the expectation that hydrogen is the only non-condensable gaseous decomposition product.

There are two significant steps of hydrogen release during the thermal decomposition of borazane in the temperature range up to 473 K performed at heating rates selected in the range of 1–5 K min^{-1}. They can be assigned to the observed two exothermic events in the DSC curves. During the first step 1.1 ± 0.1 mol H_2/mol BH_3NH_3 were released. This value is in good agreement with the proposed formation of polymeric aminoborane $(BH_2NH_2)_x$. The final value of 2.2 ± 0.1 mol H_2/mol BH_3NH_3 was reached at nearly 453 K. The amount of released hydrogen does not increase at higher temperatures and is independent of the used heating rate.

In order to obtain quantitative information about the total release of gaseous products thermogravimetric measurements were used. We have monitored the mass loss data in the temperature range up to 473 K with heating rates selected in the range of 0.5–10 K min^{-1}. The thermal decomposition of borazane is characterized by two significant steps of mass loss, which correspond to the calorimetrically detected events. Fig. 3 shows the comparison between the detected TG plots and volumetric results converted

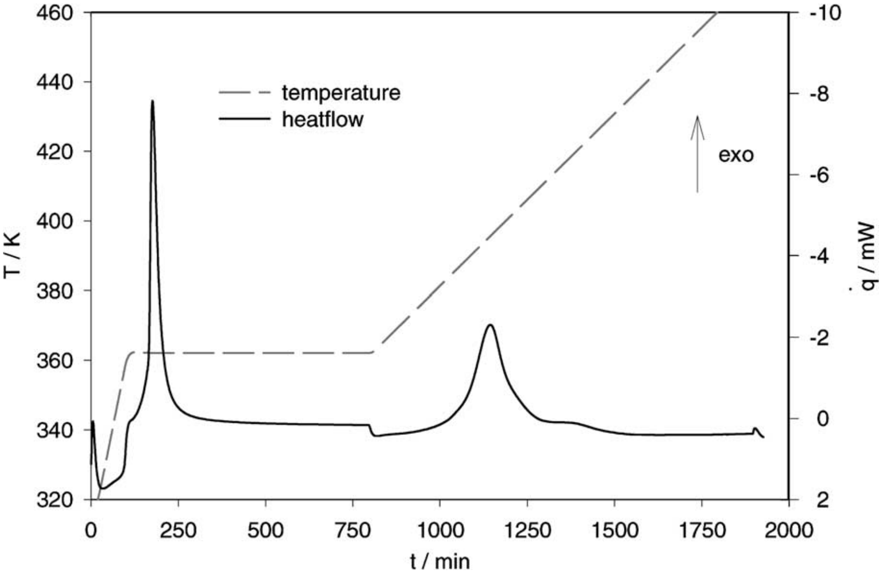

Fig. 2. Time dependence of the heat flow for the thermal decomposition of BH_3NH_3 with an isothermal part at 363 K followed by a non-isothermal part with heating rate 0.1 K min^{-1} (Setaram DSC C-80, nitrogen atmosphere).

into mass loss data. The release of 2.2 mol H_2/mol BH_3NH_3 corresponds to a mass loss of 14.3 mass% related to the mass of borazane. The comparison demonstrates the following:

1. The final amount of hydrogen released in the temperature range up to 473 K is constant ($n = 2.2 \pm 0.1$ mol H_2/mol BH_3NH_3).
2. The final mass loss obtained in TG measurements is greater than the mass of the released hydrogen. This difference increases with rising heating rate.

Obviously, during the thermal decomposition of borazane some gaseous products are released, which are condensable near room temperature, metastable or water-soluble. The amount of volatile decomposition products should increase with rising heating rate in contrast to the amount of released hydrogen, which is independent of the used heating rate. In order to identify the gaseous products the FTIR analysis of the vapors and mass spectrometric measurements are suitable.

3.3. *FTIR spectroscopic and mass spectrometric analysis of gaseous products*

Combined TG/FTIR technique allows the identification of gaseous products and their assignment to thermogravimetrically detected decomposition steps. The TG curve shows two significant steps of mass loss during the thermal decomposition of borazane in the temperature range up to 513 K performed at heating rate 0.5 K min^{-1} (Fig. 4). FTIR measurements are represented as Gram–Schmidt curve and chemigrams, which show the changes in intensities in the range 4000–400 cm^{-1} and in specific spectral regions, respectively. FTIR analysis establishes that further gaseous decomposition products are formed in addition to hydrogen, because hydrogen itself is IR-inactive. Using comparisons with known spectra data monomeric aminoborane BH_2NH_2 [11] and borazine $(BHNH)_3$ [12,13] were identified in the vapors. The formation of BH_2NH_2 and $(BHNH)_3$ was obtained during both thermogravimetrically detected decomposition steps. Monomeric aminoborane is probably unstable at room temperature and converts to non-volatile oligomers [14–16]. Pure borazine is a volatile liquid with a boiling point at 328 K. For this reason both compounds cannot be detected volumetrically. Quantitative determinations based on FTIR analysis are doubtful due to the low absorbance and overlapping spectral regions for monomeric aminoborane and borazine.

The release of gaseous products during the thermal decomposition of borazane in the temperature range

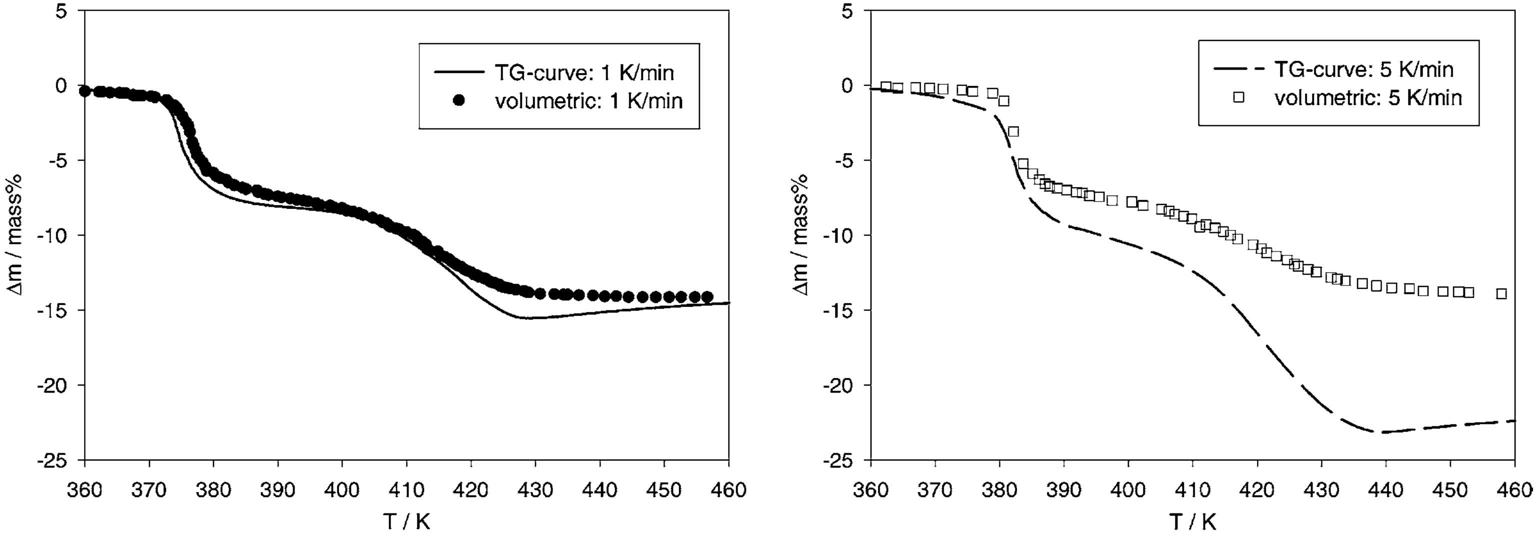

Fig. 3. Comparison between mass loss data detected thermogravimetrically (line) and calculated from volumetric results (points) (heating rate 1 K min^{-1} (a), 5 K min^{-1} (b)).

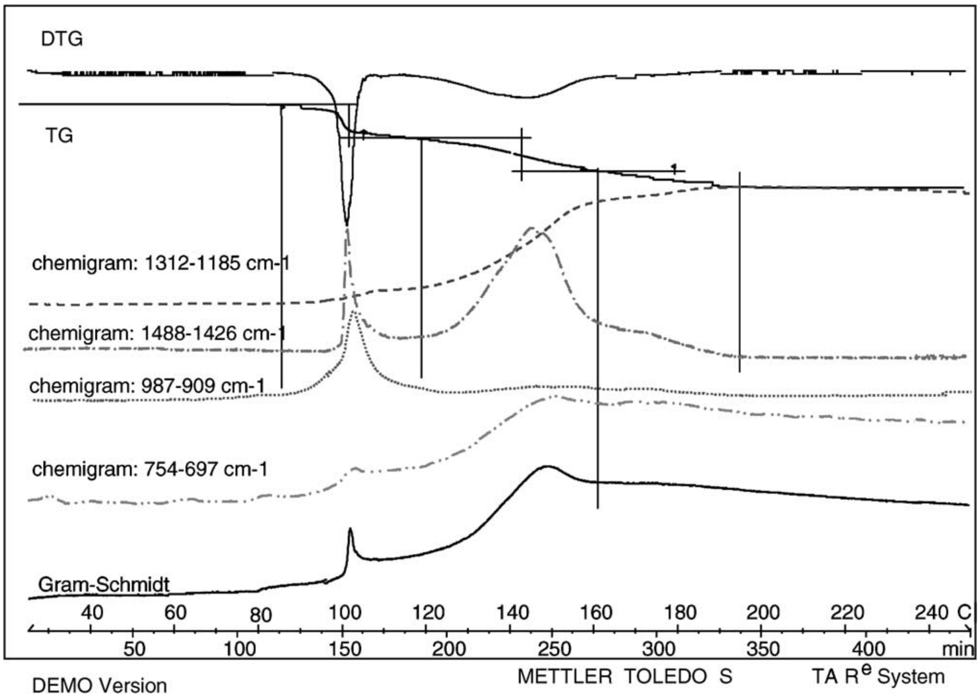

Fig. 4. Simultaneous TG/FTIR analysis of the released gas phase during the thermal decomposition of borazane (heating rate 0.5 K min^{-1}): mass loss, chemigram monomeric aminoborane, chemigram borazine, Gram–Schmidt curve.

up to 473 K was also established by means of a simultaneous TG/MS analysis. Obtained peaks at *m*23–*m*27 (Fig. 5) comply with the known mass spectrum of diborane B_2H_6 [17]. It has been reported that diborane occurs in the vapor above BH_3NH_3 [1,16,18,19]. Peaks at *m*26–*m*29 (Fig. 5) comply with the mass spectrum of monomeric aminoborane BH_2NH_2 [16]. The absence of detectable signals at

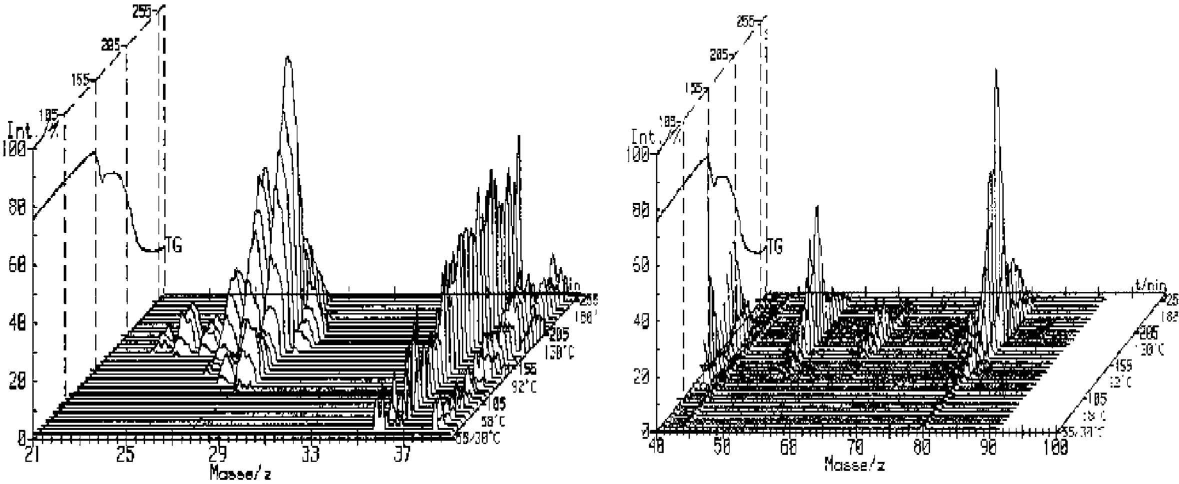

Fig. 5. Simultaneous TG/MS analysis of the released gas phase during the thermal decomposition of borazane (heating rate 1.5 K min^{-1}): mass loss, relative intensity of ion peaks at mass numbers *m*21–*m*90.

Table 1
Mass spectrum of the gas phase above BH_3NH_3 at elevated temperatures

Mass number	Assignment	Mass number	Assignment
2	H_2^+	27	$^{11}BNH_2^+, ^{10}BNH_3^+, ^{11}B_2H_5^+$
11	$^{11}B^+, ^{10}BH^+$	28	$^{11}BNH_3^+, ^{10}BNH_4^+$
12	$^{11}BH^+, ^{10}BH_2^+$	42	$^{11}BH_2NH_2{}^{11}BH_2^+$
13	$^{11}BH_2^+, ^{10}BH_3^+$	53	$^{11}B_2N_2H_3^+$
14	$^{11}BH_3^+, N^+$	62	$^{11}B_3N_2H^+$
24	$^{10}BN^+, ^{11}B_2H_2^+, ^{11}B^{10}BH_3^+$	63	$^{11}B_3N_2H_2^+$
25	$^{11}BN^+, ^{10}BNH^+, ^{11}B_2H_3^+$	78	$^{11}B_3N_3H_3^+$
26	$^{11}BNH^+, ^{10}BNH_2^+, ^{11}B_2H_4^+$	80	$^{11}B_3N_3H_5^+$

*m*30 and *m*31 indicates a negligible concentration of borazane in the gas phase. The strong signal at *m*80 refers to borazine $(BHNH)_3$ [20] (Fig. 5). No peaks were observed at mass numbers greater than *m*81. Detected mass numbers are given in Table 1 together with a possible assignment of the ion peaks.

Strong signals at *m*26–*m*29, which correspond to excited BNH_x species, have been found also in mass spectra of hydrogen-based plasma containing gaseous borazane BH_3NH_3 and borazine $(BHNH)_3$ [10]. It has been assumed [10] that excited BNH_x species act as intermediates in the CVD of c-BN. Comparison between mass spectra represented in [10] and observed in this work shows that similar gaseous species are formed during the plasma-assisted CVD process and the thermal decomposition of borazane.

On the basis of literature data recorded mass spectra were split into contributions of $(BHNH)_3$ [20], BH_2NH_2 [16], diborane [17] and hydrogen. There are small contributions to mass spectra, which cannot be assigned to these four species. The most considerable are the residual (non-assigned) ion intensities at *m*11 and *m*42. The residual intensity at *m*11 is presumably due to the inaccuracy of the used mass spectra of borazine, monomeric aminoborane or diborane (underestimated B^+ peak). The origin of the peak at *m*42 is unknown. It could be assigned to aminodiborane $BH_2NH_2BH_3$, which was reported as a product of the reaction between B_2H_6 and borazane BH_3NH_3 [21]. The peaks at *m*36 and *m*39 are due to the residual contribution of the carrier gas argon, which cannot entirely subtracted from the mass spectra.

Fig. 6 shows the temperature dependence of ion intensities corresponding to hydrogen, monomeric aminoborane and borazine at heating rate 5 K min^{-1}. Data from two independent runs are presented. Ion intensities corresponding to hydrogen were multiplied by factor 1/50 due to the high intensity level. The release of hydrogen begins at 380 K and proceeds in two steps in the temperature range up to 500 K. A clear separation of these steps is not possible at 5 K min^{-1}. The stepwise hydrogen release agrees with thermogravimetric and volumetric results. As follows from mass spectrometric analysis, BH_2NH_2 and $(BHNH)_3$ are mainly evolved during the second decomposition step in the temperature range 420–500 K. The rate of formation of these species passes through a maximum with rising temperature. At temperatures below 420 K a relatively small amount of BH_2NH_2 and borazine seems to be formed only, the main decomposition product is probably the non-volatile polymeric aminoborane $(BH_2NH_2)_x$.

It has been assumed [3] that the monomeric aminoborane may be a reactive intermediate during the formation of borazine at the thermal decomposition of borazane. Mass spectrometry supports this assumption: the release of BH_2NH_2 and borazine takes place simultaneously. Eventually borazine is formed from BH_2NH_2 via a dehydrogenation to the highly reactive monomeric iminoborane BHNH [22] and the trimerization of BHNH. But the mechanism of the formation of monomeric aminoborane BH_2NH_2 at temperatures above 400 K is unclear. BNH_x products with a lower hydrogen content are expected in this temperature range according to the detected hydrogen release. It cannot be excluded that BHNH evolves from the solid residue during the second decomposition step and undergoes two competing reactions: trimerization to borazine and hydrogenation to BH_2NH_2. The detection of small quantities of iminoborane using mass

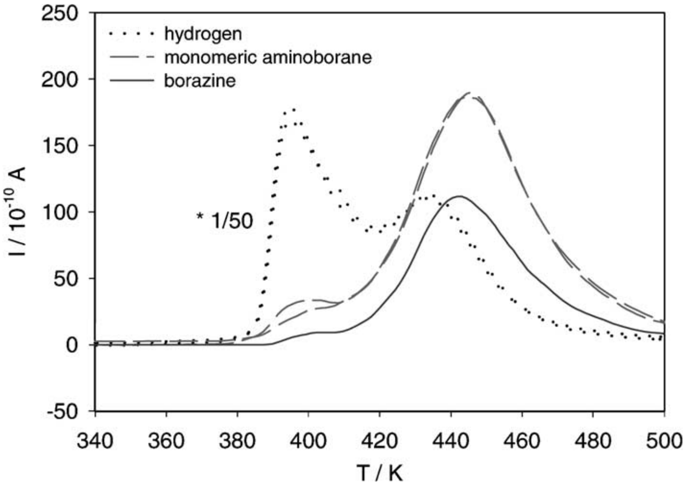

Fig. 6. Temperature dependence of the ion intensity assigned to hydrogen (points), monomeric aminoborane (– – –) and borazine (—) from two different runs (heating rate 5 K min^{-1}).

spectrometry is doubtful because of overlapping mass spectra of BHNH and BH_2NH_2.

In order to examine the influence of the heating rate on the borazane decomposition, mass spectrometric measurements are performed at heating rate 0.5 K min^{-1}. The resulting temperature dependence of ion intensities corresponding to hydrogen, monomeric aminoborane and borazine is shown in Fig. 7 (hydrogen intensities were multiplied by factor 1/50). Two steps of hydrogen release can be obtained, which agree with thermogravimetrically and volumetrically detected decomposition steps. Both steps are accompanied by the release of BH_2NH_2. In contrast, borazine was detected only during the second decomposition step (Fig. 7).

As has been shown the initial decomposition of borazane under isothermal conditions provides the clear separation of both decomposition steps. In a

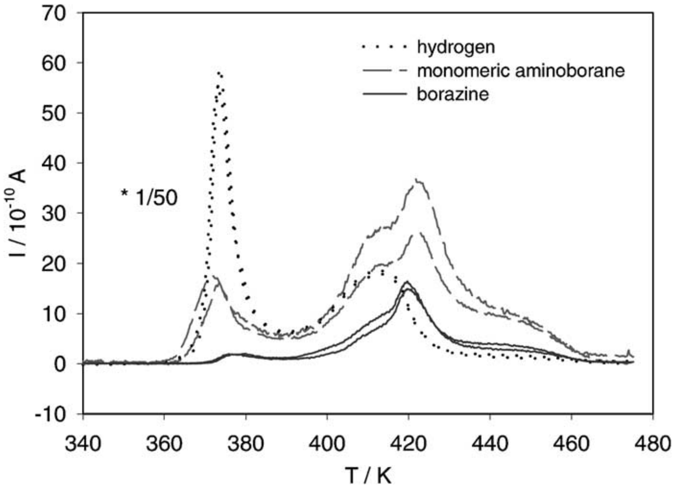

Fig. 7. Temperature dependence of the ion intensity assigned to hydrogen (points), monomeric aminoborane (– – –) and borazine (—) from two different runs (heating rate 0.5 K min^{-1}).

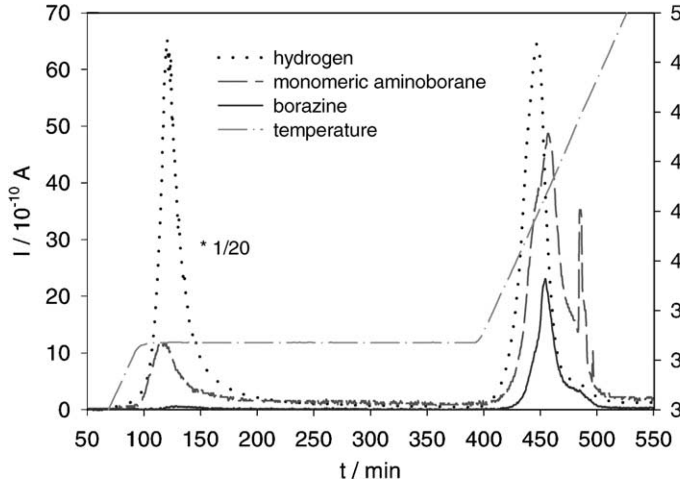

Fig. 8. Time dependence of the ion intensity assigned to hydrogen (points), monomeric aminoborane (– – –) and borazine (—) from a runs with an isothermal part at 363 K followed by a non-isothermal part with heating rate 1 K min^{-1}.

TG/MS run borazane was held 5 h at 363 K and then heated up to 550 K at heating rate 1 K min^{-1}. The time dependence of the ion intensities corresponding to hydrogen, monomeric aminoborane and borazine is shown in Fig. 8. The ion intensity corresponding to hydrogen was multiplied by factor 1/20. At 363 K hydrogen and monomeric aminoborane were detected in the gas phase. The amount of borazine released at 363 K is negligible. The formation of hydrogen and BH_2NH_2 at 363 K terminates after more than 3 h. Mass spectrometric results establish that the degradation of the solid polymeric aminoborane $(BH_2NH_2)_x$ under formation of gaseous monomeric aminoborane is insignificant at 363 K. With increasing temperature an additional mass loss was observed, which can be assigned to the second decomposition step. The second decomposition step is accompanied by the formation of hydrogen, monomeric aminoborane and borazine (Fig. 8). The formation of borazine takes place at temperatures above 400 K.

The quantitative analysis of gaseous products on the basis of mass spectrometric data is questionable. Detected ion intensities really provide a measure of the frequency of the different species, but with increased mass of species the permeability of the coupling system and the mass spectrometer is reduced. Thus, more excessive intensity values are recorded for light species. In order to obtain quantitative results a calibration with pure decomposition products would be required, e.g. by Pulse Thermal Analysis [23]. Only a rough assessment of the amount of gaseous decomposition products is possible now. We have approximated TG plots using the temperature dependence of integrated mass spectra of hydrogen, monomeric aminoborane, borazine and diborane. According to volumetric results the release of 2.2 mol H_2/mol BH_3NH_3 up to the final temperature of 500 K was assumed. Calculation results for different runs are given in Table 2. Monomeric aminoborane appears to be the main impurity of the released hydrogen. It seems that the yield of borazine and BH_2NH_2 increases with rising heating rate. But it should be noted that this is only a semi-quantitative assessment of the amount of decomposition products.

Table 2
Yield of gaseous products of the thermal decomposition of borazane at temperatures up to 500 K calculated on the basis of thermogravimetric and mass spectrometric data for different runs

Heating rate	Product yield (mol product per mole borazane)			
	H_2	BH_2NH_2	Borazine	Diborane
0.5 K min^{-1}	2.2	0.12	0.035	0.02
1.5 K min^{-1}	2.2	0.16	0.05	0.025
5 K min^{-1}	2.2	0.21	0.065	0.04
Iso 363 K–1 K min^{-1}	2.2	0.12	0.04	0.02

4. Conclusions

Borazane BH_3NH_3 undergoes stepwise thermal decomposition in the temperature range up to 500 K. DSC measurements at high heating rates show two partially overlapping exothermic events assigned to two decomposition steps and an endothermic event assigned to the melting of borazane. Both decomposition steps can be separated, if the first step is realized under isothermal conditions at temperatures below 363 K. The exothermic decomposition steps are accompanied by a stepwise hydrogen release. Approximately 2.2 mol H_2/mol BH_3NH_3 were formed up to 473 K independent of the heating rate. This corresponds to a hydrogen storage density of 14.3 mass% related to the mass of borazane. The measured mass loss is greater than the mass loss caused by hydrogen release. Consequently, in addition to hydrogen some further gaseous products must be formed during the thermal decomposition of borazane. The final mass loss is increased from 14 to 33 mass% when the heating rate is increased from 0.5 to 10 K min^{-1}.

The formation of gaseous monomeric aminoborane BH_2NH_2 and borazine $(BHNH)_3$ during the thermal decomposition of borazane was proved by means of mass spectrometry and FTIR spectroscopy. Additionally, small quantities of diborane B_2H_6 are released as follows from mass spectrometric analysis. Borazine was released only at temperatures above 400 K. The estimated yield of borazine grows with rising heating rate. The influence of the thermal history on the borazine formation appears to be insignificant. Mass spectrometric data supports the assumption that borazine is formed via the intermediate monomeric aminoborane.

Obtained mass spectra show some similarities to data, which was recently reported for hydrogen-based plasma containing gaseous borazane and borazine. In both cases mass spectra contain signals assigned to BNH_x species. It is possible that excited BNH_x species play an important role in the plasma-assisted CVD of c-BN. Further investigations of gas-phase reactions during the thermal decomposition of borazane could be of interest due to a conceivable deposition of boron nitride layers.

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