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Nanosized Si–C–N-Powders by polysilazane pyrolysis and Si_3N_4/SiC-Composite Materials Thereof

G. Boden,[a]* A. Neumann,[a] T. Breuning,[a] E. Tschernikova[b] and W. Hermel[a]

[a]Fraunhofer-Institute for Ceramic Technologies and Sintered Materials (IKTS), Dresden, Germany
[b]Institute for Problems of Material Sciences, Kiev, Ukraine

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Abstract

Nanosized silicon carbonitride powders have been synthesized by gas phase pyrolysis of evaporable precursors at temperatures between 600 and 1000°C. These synthesis have been carried out by using of mixtures of cyclic polysilazanes, which were prepared by ammonolysis of dimethyldichlorosilane in an aprotic organic solvent, or of hexamethyidisilazane as starting materials. The spherical powder particles are X-amorphous. They demonstrate a particle size from 100 to 600 nm and a specific surface area from 10 to 350 m^2/g. The Si–C–N-powders crystallize upper 1400°C to Si_3N_4- and SiC-phases. The chemical composition, the specific surface area and the particle size distribution of the Si–C–N-powders are controlled by changing the starting materials, the synthesis and the pyrolysis conditions. The carbon content of the powders consists of bonded carbon in the SiC and of free carbon. By pyrolysis or post heating in reactive ammonia the carbon content can be diminished. On-line coupling of thermogravimetry with evolved gas analysis/mass spectrometry have been applied to characterize the behaviour of powders during annealing processes. The investigations indicate, that after the pyrolysis organic species are adsorbed onto the surface of the powders, which can be removed by suitable post-treatment. From the synthesized Si–C–N-powders Si_3N_4/SiC-composite materials have been obtained by sintering. In these composites SiC nanograins are arranged both in the intra- and in the inter-type and hinder the growth of the Si_3N_4-grains.

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1 Introduction

Polysilazanes have a wide range of applications for synthesis of different silicon carbonitride materials. At first, curable polysilazane precursors have been used for materials with a great ratio of surface to volume, all above for fibers[1–7] and coatings[8] and as binder.[9–12] Thereby the melt or the viscous solution of the polysilazane is formed into the desirable arrangement (mainly fiber, but also thin layer, coating or binder), cross-linked and pyrolyzed at temperatures above 1000–1200°C. An intrinsic problem of this procedure is the shrinkage of the polysilazane during pyrolysis and the difficulty to get dense materials.

Secondly polysilazanes and silicon carbonitride powders can be prepared by milling of a curable polysilazane or of the pyrolyzed silicon carbonitride powder thereof.[13–18] Thereby different variants can be used. Or the cured polysilazane is pyrolyzed and milled or the polysilazane is mixed with a silicon carbonitride or a silicon nitride powder and this mixture is milled. The particle size of these milled silicon carbonitride powders, synthesized by polysilazane pyrolysis or in presence of polysilazenes, is limited by the milling aggregate and amounts to 0·8–1 μm. This size of the powder particles is not sufficient and not advantageous to reaching a nano-structured Si_3N_4/SiC-composite material. For such nano-structured composite materials nanosized silicon carbonitride powders are necessary, which can be prepared, thirdly, by gas phase-pyrolysis of evaporable oligo- or polysilazanes or amino-substituated silanes.

The most used silazane for gas phase pyrolysis is hexamethyldisilazane. This commercially available product is easily evaporable (boiling point: 126°C) and streams, often by means of an inert gas, into the pyrolysis equipment, which can be a heated

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*To whom correspondence should be addressed. Fax: 0049 3 512553600; e-mail: boden@ikts.fhg.de

tube[19–23] or laser equipment.[24–28] Hexamethyldisilazane possesses a great ratio of carbon to nitrogen, based on the six methyl-groups at the two silicon atoms. Therefore the content of carbon (bonded and free) in the silicon carbonitride powder, is often too high. Besides hexamethyldisilazane further defined cyclic silazanes, e.g. trimethylcyclotrisilazane and tetramethylcyclotetrasilazane[29–32] and tris(dimethylamino)silan[33] have been used as starting materials for the synthesis of silicon carbonitride. Alternatively to the use of small single molecules, which contain only one ceramic generating element (SiH_4, NH_3, C_xH_y) for the co-pyrolysis, the silazanes are very advantageous for the preparation of homogeneous silicon carbonitride powders, because the –C–Si–N-groups are preformed and reach at the pyrolysis a homogeneous network without local enrichments of one ceramic generating element.

Finally should be mentioned the preparation of nanosized silicon carbonitride powder by pyrolysis of a highly crosslinked silylcarbodiimide,[34] which has been synthesized from silicon tetrachloride and *bis*(trimethylsilyl)carbodiimide in an organic solvent.

Such nanosized silicon carbonitride powders can be used for preparing Si_3N_4/SiC-nanocomposite materials to improve the properties of Si_3N_4 ceramics by dispersing second phases. One way is the intrinsic incorporation of SiC grains into the Si_3N_4 matrix. Niihara and co-workers[19–23,35] used silicon carbonitride powder pyrolyzed from hexamethyldisilazane and prepared Si_3N_4/SiC composite materials by hot pressing. They developed the theoretical concept of nanocomposites and proved on practical results. They found a good accordance. In this concept nanograins of SiC will be introduced intergranularly and/or intragranularly between and/or into the Si_3N_4 micro grains, respectively. In most cases an improvement of the fracture toughness and the bending strength was found for 5–34 vol% SiC.[36]

Recently Mayne *et al.*[37] used mixtures of silicon nitride and nanoscaled silicon carbonitride powders (particle size 30–60 nm, produced by laser synthesis) to prepare Si_3N_4/SiC-composite materials by hot pressing. Thereby a fine microstructure with a tendency to plastic deformation (high toughness) has been observed.

Lences *et al.*[38] prepared Si/C/N-materials from as-received Si/C/N-powders (particle size 15–20 nm, produced in a CO_2-laser) and observed a very fine microstructure in the dense samples.

Below will be reported a new way of preparation of silicon carbonitride powders by precursor pyrolysis. Further hexamethyldisilazane (HMDS) pyrolysis is used to compare the obtained results.

2 Preparation of the Si–C–N-Powders

2.1 Synthesis of the polysilazane precursors

The first step for the preparation of silicon carbonitride powders is the synthesis of volatile polysilazanes. The polysilazanes have been synthesized by reaction of dimethyldichlorosilane with dried gaseous ammonia in an aprotic organic solvent (dichloromethane, toluene, tetrahydrofuran). Brewer and Haber[39] described the synthesis of polymethylcyclopolysilazanes both by solvent ammonolysis in dry benzene and by direct ammonolysis in liquid ammonia. In our investigations the ammonolysis in the non-flammable solvent dichloromethane has been favoured. There have been used 2- and 4-l flasks with 1 or 2 mol (129 g or 258 g) dimethyldichlorosilane respectively in 1·5 or 3 l dichloromethane. In one attempt 60 g and 120 g polysilazane respectively were obtained. This corresponds to a chemical yield of nearly 80%. Figure 1 shows the scheme of the synthesis apparatus.

In the reaction above between dimethyldichlorosilane and ammonia all ring shaped polysilazanes have been prepared [eqn (1)].

$$n\mathrm{Si(CH_3)_2Cl_2} + 3n\mathrm{NH_3} \rightarrow [\mathrm{Si(CH_3)_2NH}]_n + 2n\mathrm{NH_4Cl} \qquad n = 3-4 \tag{1}$$

The chemical reaction (1) is followed by the filtration of the NH_4Cl, the distillation of the solvent and the vacuum distillation of the polysilazane mixture. The last is not necessary, if the polysilazane mixture is directly pyrolyzed. It is necessary to realize the synthesis of the polysilazanes in the absence of oxygen and moisture. Therefore a dry stream of nitrogen during all operations is used.

The synthesized mixture of polysilazanes is a liquid–waxy to solid, colourless to yellowish substance. In Table 1 the physical properties and the formula of one synthesized mixture is reported in comparison to defined oligosilazanes. Table 2 summarizes the composition of the synthesized polysilazanes.

The mixture of the synthesized polysilazanes mainly consists of liquid hexamethylcyclotrisilazane (HMCTS) and of solid octamethylcyclotetrasilazane

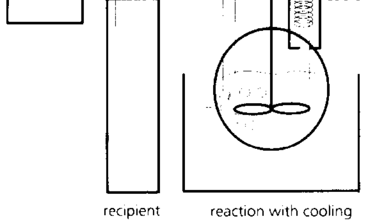

Fig. 1. Scheme of the apparatus for the synthesis of the polysilazane mixture from dimethyldichlorosilane and ammonia.

Table 1. Physical properties and formula of evaporated silazanes

	Formula	*Mol-weight (g)*	*Density (g/cm³)*	*Melting point (°C)*	*Boiling point (°C)*
Hexamethylcyclotrisilazane	$C_6H_{21}N_3Si_3$	220	0·920	−10	186
Octamethylcyclotetrasilazane	$C_8H_{28}N_4Si_4$	293	0·950	97	225
Hexamethyldisilazane	$C_6H_{19}N\ Si_2$	161	0·765	−80	126
Synth· mixture	$Si_1N_{0\cdot93}C_{1\cdot84}H_{4\cdot9}O_{0\cdot07}$ $=C_1H_{2\cdot67}N_{0\cdot5}Si_{0\cdot54}O_{0\cdot04}$		0·93	55...90	90...250

(OMCTS). Nearly 5–10% short-chaining oligosilazanes have been obtained, which are not evaporated but cross-linked. After evaporation of the cyclic oligosilazanes a brown paste-like residue remains in the evaporating vessel.

In Fig. 2 the DRIFT-spectrum of a synthesized polysilazane is shown and compared with pure HMCTS and OMCTS. It appears, that the synthesized product consists of a mixture of HMCTS and OMCTS.

2.2 Pyrolysis of the volatile polysilazane mixture

In a second step the pyrolysis of the synthesized polysilazane mixture has been realized according to eqn (2)

$$[Si(CH_3)_2NH]_n \rightarrow [Si - C - N]_y + H_2/CH_4/C + NH_3 \qquad y \gg n \tag{2}$$

Thereby the vapour of the polysilazanes has been streaming by a dry inert (N_2) or reactive (NH_3) gas or mixtures through a heated tube (800–1100°C). The scheme of the pyrolysis equipment is shown in Fig. 3. The tube has a diameter of 30 mm, and the length is 500 mm, of which the length of the heated zone is 300 mm. Every run of a synthesis gives 5 g Si–C–N-powder. (For comparison: the HMDS-pyrolysis that is realized in a tube with 55 mm diameter and 600 mm length, 50 g Si–C–N-powder is prepared).

The heating tube can be arranged in a horizontal or vertical direction. In both cases the conditions of the pyrolysis (temperature, carrier gas composition, precursor content in the carrier gas stream) are comparable. In the vertical arrangement the feeding of the gas and precursor mixture is possible from the top or the bottom.

The silicon carbonitride powder is deposited in the heating tube and in the deposition chamber with filter candle. About this a small quantity (near 5%) of silicon carbonitride is deposited as a thin and hard layer at the beginning of the heating tube. This product adheres to the inner wall of the tube and may not be mixed with the fine grained Si–C–N-powder.

The ceramic yield reaches nearly 50%, both for the mixture of cyclic polysilazanes and for the isolated HMCTS or OMCTS.

Table 2. Composition of the synthesized polysilazanes (in wt%)

	Theoretical value for silazane unit	*Experimental values (range)*	*Average value*
Si	38·4	35–40	37·5
N	19·2	15–20	17·5
C	32·8	25–32	28·5
O	0	0·5–3	1·5
H	9·6	5–8	6·5
N:C	1:2	1:2	1:2

After the pyrolysis the Si–C–N-powders were annealed at 1350°C for 2 h. Thereby the powders lose residue-groups and radicals (e.g. CH_x, NH_y) from the surface, but do not yet crystallize.

3 Properties of the Synthesized Si–C–N-Powders

3.1 Composition of the polysilazane pyrolyzed Si–C–N-powders

The composition of the precursor pyrolyzed Si–C–N-powders depends on the pyrolysis atmosphere. The carbon content of the powder consists of bonded carbon in the SiC and free carbon. The pyrolysis in the inert nitrogen stream leads to carbon-rich

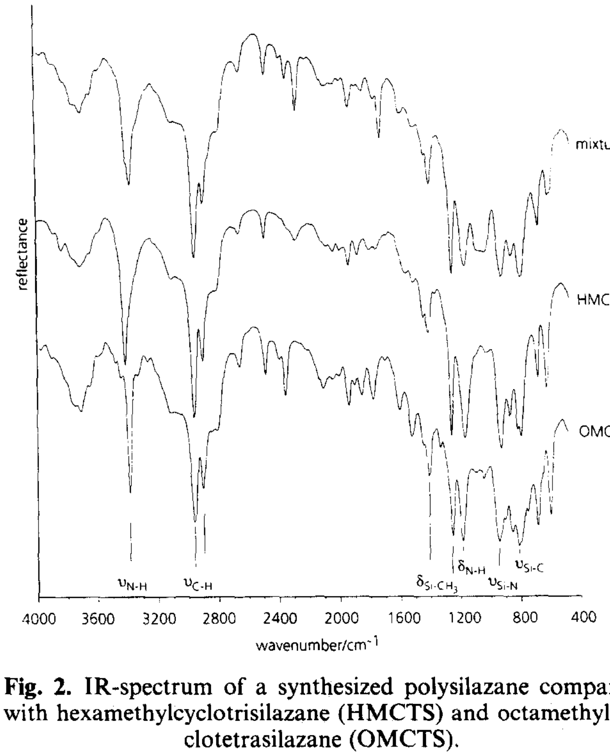

Fig. 2. IR-spectrum of a synthesized polysilazane compared with hexamethylcyclotrisilazane (HMCTS) and octamethylcyclotetrasilazane (OMCTS).

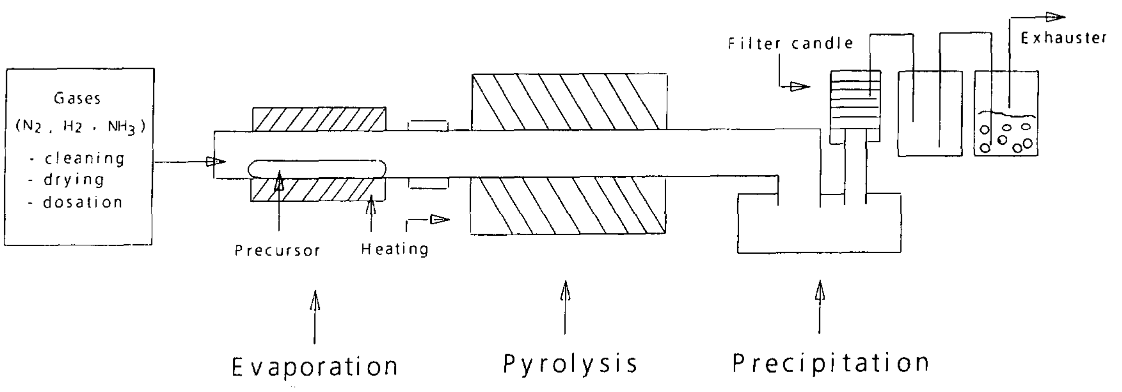

Fig. 3. Scheme of the pyrolysis equipment for the synthesis of Si–C–N-powders.

powders, which contain free carbon of up to 10–20%. By pyrolysis in the reactive ammonia nitrogen-rich powders without or with a small quantity of free carbon have been prepared. Free carbon can also be decreased by post-treatment in ammonia or hydrogen/nitrogen gas (1:1) at 1350°C.

In Table 3 and Fig. 4 the composition of the polysilazane pyrolyzed silicon carbonitride powders is summarized.

The synthesized Si–C–N-powders exhibit a high purity. The quantity of metallic impurities (Fe, Al, Ca, Mg, Na) does not exceed 300 ppm. The content of chlorine amounts to 150 ppm and decreases after treatment at 1500°C, to 20 ppm.

The content of oxygen in the Si–C–N-powders increases from 1·0 to 1·5 wt% immediately after the synthesis to 5–8 wt% at handling on air. The powders are very sensitive to oxygen and moisture and partially pyrophorous. Thus the handling under inert atmosphere in glove boxes is recommended.

3.2 Composition of the HMDS-pyrolyzed Si–C–N-powders

In comparison to the silicon carbonitride powders obtained from polysilazane pyrolysis, silicon carbonitride powders pyrolyzed from HMDS have been investigated. HMDS is a relatively cheap commercial product and is therefore a good base for the preparation of Si–C–N-powders in large quantities. The disadvantage of HDMS is the great ratio carbon to nitrogen (6:1) in comparison to the cyclic polysilazanes. In these the ratio carbon to nitrogen is 2:1. For that reason the HMDS-pyrolyzed Si–C–N-powders (inert nitrogen pyrolysis) have a higher content of carbon and a lower content of nitrogen than the polysilazane-pyrolyzed Si–C–N-powders. In Table 4 the results of the HMDS-pyrolysis are compared with those of the polysilazane pyrolysis under the same conditions.

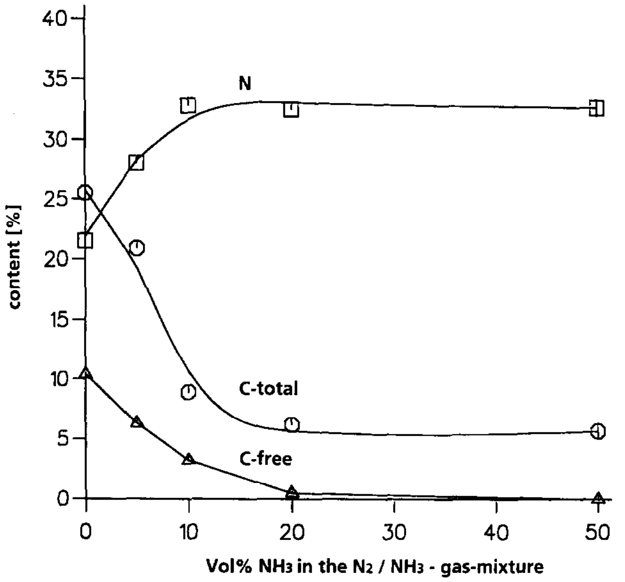

Fig. 4. Relations between composition of the pyrolysis gas and the resulting Si–C–N-powder, pyrolyzed from polysilazane mixture.

Table 3. Composition of the Si–C–N-powders in dependence of the pyrolysis atmosphere

Weight %	*Si–C–N-powder*	
	Pyrolysis in N_2 or Ar	*Pyrolysis in NH_3*
Si	40–48	50–56
N	18–22	32–35
C	24–28	0·5–5
O	2–3	2–4
H	< 0·1	< 0·1
N:C	1:1·5	

Based on the relatively great quantity of outgassing groups at the pyrolysis of HMDS a low ceramic yield is expected. According to this we find, that in the HMDS-pyrolysis the ceramic yield reaches 38–40%.

3.3 Further properties of the Si–C–N-powders

The powders possess a brown to black colour after synthesis in an inert atmosphere, but in reactive ammonia atmosphere the colour turns from light brown to white. In Table 5 further properties of the powders are summarized.

Table 4. Carbon and nitrogen contents of Si–C–N powders, pyrolyzed from different polysilazanes (950°C, N_2-stream 20 l/h, tube diameter 30 mm)

Silazane	*Content (wt%)*	
	Carbon	*Nitrogen*
HMDS	33·2	12·5
HMCTS	22·7	22·1
OMCTS	23·6	22·8
Polysilazane	25·5	22·0

The spherical powder particles agglomerate. The primary particles have a diameter from 100 to 600 nm.

In Fig. 5 the SEM-photography of a Si–C–N-powder pyrolyzed from polysilazane has been shown. The Si–C–N-powders have been amorphous after the synthesis and heat post-treatment at 1350°C, but they crystallize at temperatures above 1400°C to Si_3N_4- and SiC-phases. X-ray-analysis showed that powders without free carbon partitioned to α-Si_3N_4 and β-Si_3N_4, but not crystalline SiC-phases. Powders containing free carbon crystallize to α-Si_3N_4 and β-SiC. Obviously, the free carbon prevents the formation of β-Si_3N_4 and induced the crystallization of the amorphous SiC to β-SiC. Figure 6 shows Si–C–N-powder crystallized at 1500°C. A part of the powder is amorphous and equiaxed but single whisker-like α-Si_3N_4-crystals grow out of the powder agglomerates.[40]

4 Investigations on the Thermal Behaviour of the Si–C–N-Powders (HMDS-pyrolysis)

The simulation of the behaviour of the Si–C–N-powders during annealing processes have been performed by thermogravimetry–mass spectroscopy (TG–MS) analysis using a Netzsch STA 429 apparatus on-line coupled with a Balzers QMG 421 quadrupol mass spectrometer, working under a dynamic nitrogen atmosphere (99·999% purity) at a flow rate of 66·7 ml/min. The results demonstrate, that the amount of the adsorbed species on the synthesized powders depends on the pyrolysis conditions, such as temperature and atmosphere.

Table 5. Properties of the polysilazane pyrolyzed Si–C–N-powders

Colour	*Black (inert gas) to light brown (reactive gas)*
Morphology	*Spherical particles, agglomerated*
Density	0·1 g/cm^3
Particle size	100–600 nm
BET-surface after synthesis	40–350 m^2/g
after heat treatment at 1350°C	10–50 m^2/g
Phases	Amorphous

Fig. 5. SEM-photography of the synthesized Si–C–N-powders.

Fig. 6. SEM-photography of Si–C–N-powder heated at 1500°C (crystallization to α-Si_3N_4).

The influence of pyrolysis temperature is clearly demonstrated in Fig. 7. An increased pyrolysis temperature leads to a decrease of the adsorbed species and therefore the mass loss is lower.

Figure 8 shows the mass change versus the composition of pyrolysis atmosphere.

The greater mass loss of powder pyrolyzed under ammonia is probably due to a higher amount of adsorbed species (up to 900°C) as well as the reaction of included oxygen with the free carbon or the carbon of the framework at higher temperatures.[41]

The TG–MS investigations (Fig. 9) demonstrate that the TG curve may be distinguished in three main regions. Between room temperature and 400°C a 0·8% mass loss is measured. The mass spectrometric investigations indicate a release of water (in the form of OH m/e 17; m = mass; e = charge), benzene (m/e 78) and traces of silicon compounds. Mass ions for the silicon compounds suggest the existence of disilazane compounds $(CH_3SiH_2)_2NH$ (m/e 105) and $(CH_3)_3SiNHSiH(CH_3)_2$ (m/e 147), which are probably physisorbed.

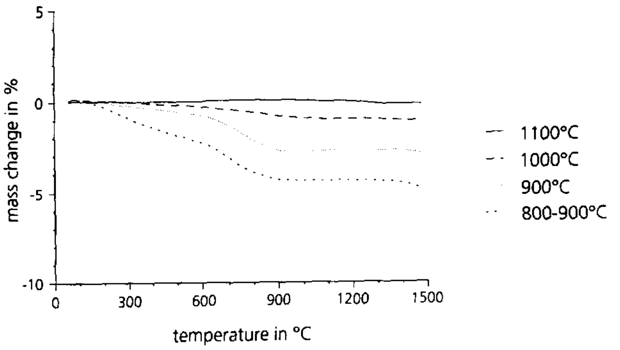

Fig. 7. Mass change of Si–C–N-powder obtained from HMDS-pyrolysis under nitrogen at different temperatures.

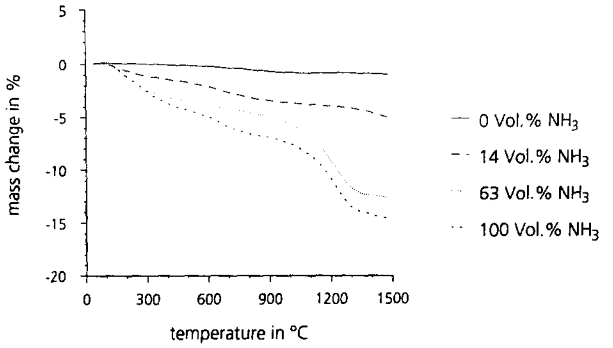

Fig. 8. Mass change of Si–C–N-powder obtained from HMDS-pyrolysis at 1000°C under different N_2/NH_3-mixtures.

The formation of benzene during pyrolysis is possible through reactions similar to the catalytic reforming process (transformation of aliphatic to aromatic hydrocarbons). Water may arised by condensation of OH-groups or by reaction of hydrogen and oxygen at higher temperatures. The major mass loss of 3 % occurs between 400 and 1100°C and corresponds mainly to an escape of hydrogen (*m/e* 2), water and methane (*m/e* 16), but also mass ions suggesting the existence of hydrocarbons (acetylene, ethene) are detected. It is assumed that these compounds are chemisorbed onto the powder. A small mass loss of 0·4% which corresponds to a release of carbon monoxide (*m/e* 12) is measured above 1100°C. At higher temperatures the oxygen included in the powder reacts either with the free carbon or the carbon of the framework.

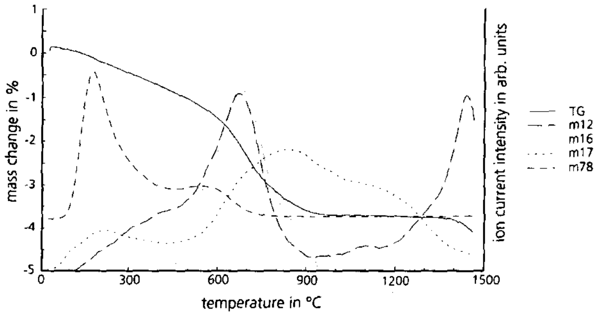

Fig. 9. Mass change and ion current intensities of characteristic mass numbers of the Si–C–N-powder obtained from HMDS-pyrolysis at 800–900°C under nitrogen.

The results of the on-line coupled TG–MS investigations has led to the following conclusions: the powder synthesis by pyrolysis from silazanes should be realized at temperatures between 1000 and 1100°C or the synthesized powders must be post-treated at temperatures higher than 1200°C.

Conclusions for the mechanism of the silazane pyrolysis can be obtained. Possible steps of the formation of the Si–C–N-powders from –C–Si–N-units are demonstrated in Fig. 10.

It can be seen that at 800–1100°C physisorbed (CH_4, NH_3) and chemically bonded groups ($-CH_3$, $-NH_2$) containing hydrogen are arranged on the powder and the surface. These groups disappear by subsequent heat-treatment. Above 1400°C crystallization takes place.

5 Si_3N_4/SiC-Composite Materials from the Synthesized Si–C–N-Powders

Nanostructured Si_3N_4/SiC-composite materials combine the properties of silicon carbide and silicon nitride materials, the excellent high temperature creep and the relatively good toughness.[21] Therefore the nano-grains of SiC in the Si_3N_4 matrix can be arranged intergranularly and intragranularly.[35]

5.1 Si_3N_4/SiC-composites from polysilazane pyrolyzed Si–C–N-powder

Si–C–N-powder, synthesized by pyrolysis of a polysilazane mixture, used for preparation of a composite material, contains 35% SiC (11·5% C; 25·5% N; 2·4%). This powder has been mixed in isopropanol by a ball mill together with 8% Y_2O_3 as sintering aid, dried in a vacuum rotation evaporator and hot pressed (1800°C, 30 MPa, 1 h). Thereby dense composite materials have been obtained.

In Fig. 11 the microstructure of the composite material is shown. All above intergranular nano-SiC grains are situated between the Si_3N_4 micrograins. About this the Si_3N_4-grains contain intragranular SiC-grains, comparable with the results of Sajgalik *et al.*, [42] who reported on TEM-investigations on SiC/Si_3N_4 nano/micro composites prepared from a mixture of Si_3N_4 and 10% amorphous Si–C–N-powder. Sawaguchi *et al.*[43] obtained similar results. Thereby a Si_3N_4/SiC nanocomposite was prepared from HMDS-pyrolyzed Si–C–N-powder. The TEM image of the composite shows distributed nanosized SiC-particles in the Si_3N_4 matrix (intragranular) at low concentration of SiC in the powder and on the grain boundaries (intergranular) at higher content of SiC in the powder.

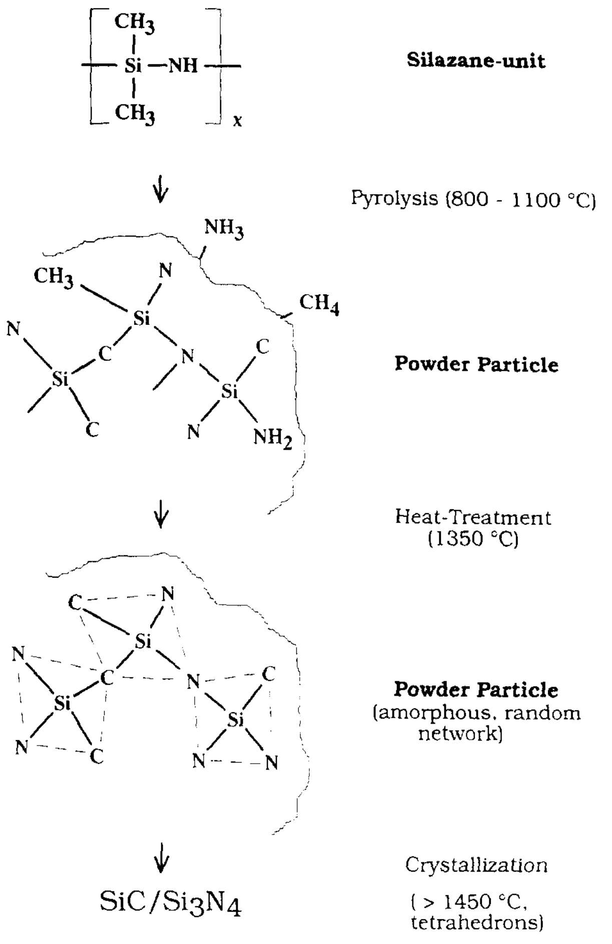

Fig. 10. Possible steps of the formation of Si–C–N-powders.

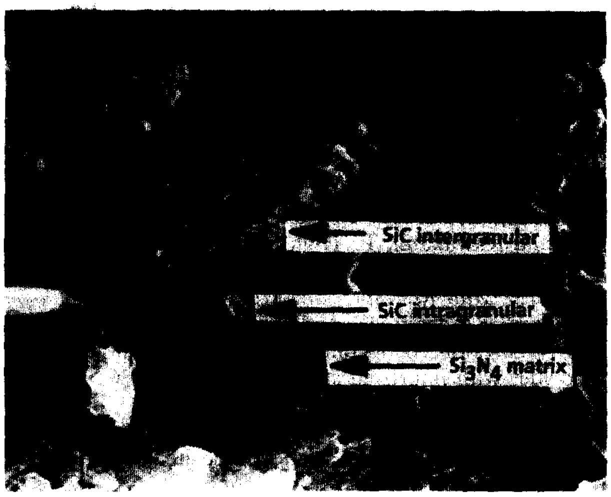

Fig. 11. SEM-photography of Si_3N_4/SiC-composite prepared from polysilazane pyrolyzed Si–C–N-powder.

5.2 Si_3N_4/SiC-composites from HMDS-pyrolyzed Si–C–N-powder

For the preparation of Si_3N_4/SiC-composite materials mixtures of commercial α-Si_3N_4-powder with up to 43 wt% synthesized Si–C–N-powder (HMDS pyrolysis) and sintering additives have been used as starting materials. The silicon carbonitride powder contains nearly 30% SiC (10·5% C; 32·1% N; 2·3% O). The powder mixtures have been hot pressed (1840°C, 30 MPa, 1 h). The mechanical properties of the composite materials are summarized in Table 6.[44]

A fully densified Si_3N_4-SiC-nanocomposite can be obtained by addition of amorphous Si–C–N powder synthesized by gas phase pyrolysis of HMDS to a starting mixture of conventional crystalline Si_3N_4 powder and sintering additives following hot pressing (Table 6). Thereby the hardness of the sintered composite materials increases with increasing content of silicon carbonitride powder. The toughness of the composites decreases with increasing content of SiC in the mixture in the same way.

Figure 12 shows the structure of a hot-pressed Si_3N_4/SiC nano-composite material (LN2).

There have been created nanocomposites with SiC-nanograins inside the Si_3N_4-grains and between the Si_3N_4-grains. SEM micrographs show that the silicon carbide particles are finer than 50 nm in diameter. The nanostructure in the composite has been observed beginning with a content of 5 wt% Si–C–N-powder. The growth of elongated Si_3N_4-grains decreased with the increase of SiC content, and successive equiaxed and fine Si_3N_4 grains have been developed.

The presence of SiC grains at the grain boundary hinder the growth of large β-Si_3N_4. The change in the microstructure can be explained also in an other way: with increasing SiC-content also an increasing amount of another kind of Si_3N_4 source,

Table 6. Composition and properties of sintered (HP) Si_3N_4/SiC-composites

Name	*Y_2O_3 (wt%)*	*Al_2O_3 (wt%)*	*Si–C–N (wt%)*	*D (g/cm^3)*	*HV10 (GPa)*	*K_{IC}[a] ($MPa\ m^{1/2}$)*	*K_{IC}[b] ($MPa\ m^{1/2}$)*
LIA	1·8	3	—	3·186	15·3	7·2	3·7
LN1	1·8	3	1	3·193	15·6	6·3	3·8
LN2	1·8	3	5	3·197	15·2	6·3	3·8
LN3	1·8	3	30	3·185	16·4	4·3	3·3
LN4	1·8	3	43	3·213	16·6	4·4	3·2

[a] SENB method; [b] ISB-method (see Ref. 44).

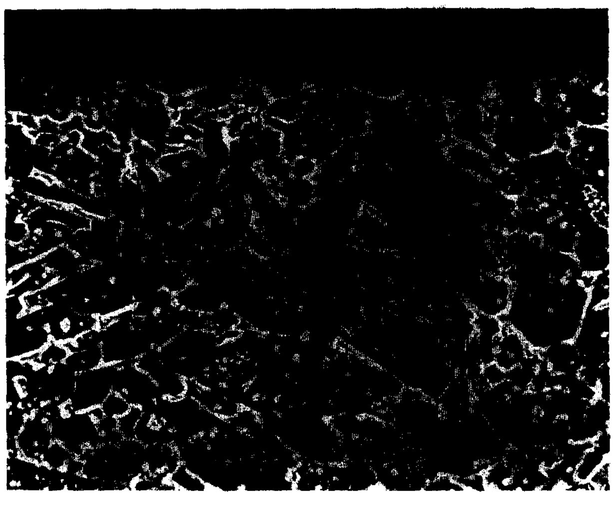

Fig. 12. SEM-photography of Si_3N_4/SiC-composite produced with 5% Si–C–N-powder.

produced by precursor pyrolysis is introduced in the materials (31% in the material LN4). This may influence the microstructure.

6 Conclusions

The synthesized mixture of evaporable cyclic polysilazanes, which has been prepared by ammonoiysis of dimethyldichlorosilane, is a suitable starting material for homogeneous nano/micro grained silicon carbonitride powder. About this the cyclic polysilazanes yield a lower content of carbon in the Si–C–N-powders than the dimer hexamethyldisilazane. Generally the composition of the Si–C–N-powders depends on the pyrolysis atmosphere: in inert atmosphere carbon-rich powders have been generated, but with increasing content of the reactive ammonia gas in the pyrolysis atmosphere the content of nitrogen in the powder rises.

From the synthesized Si–C–N-powders Si_3N_4/SiC-composite materials, which contain inclusions of nano-SiC grains both in the intra-type and in the inter-type, can be obtained by hot pressing.

For the preparation of a Si_3N_4/SiC-composite material with excellent mechanical properties a steady feedback between the parameters of the powder synthesis, the properties of the Si–C–N-powders and the composite material is necessary.

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