

# Influence of the $\alpha/\beta$ -SiC phase transformation on microstructural development and mechanical properties of liquid phase sintered silicon carbide

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The transformation kinetics and microstructural development of liquid phase sintered silicon carbide ceramics (LPS-SiC) are investigated. Complete densification is achieved by pressureless and gas pressure sintering in argon and nitrogen atmospheres with  $Y_2O_3$  and AlN as sintering additives. Studies of the phase transformation from  $\beta$  to  $\alpha$ -SiC reveals a dependency on the initial  $\beta$ -content and the sintering atmosphere. The transformation rate decreases with an increasing  $\beta$ -content in the starting powder and in presence of nitrogen. The transformation is completely suppressed for pure  $\beta$ -SiC starting powders when the additive system consists of 10.34 wt %  $Y_2O_3$  and 2.95 wt % AlN. Materials without phase transformation showed a homogeneous microstructure with equiaxed grains, whereas microstructures with elongated grains were developed from SiC powders with a high initial  $\alpha/\beta$ -ratio ( $> 1:9$ ) when phase transformation occurs. Since liquid phase sintered silicon carbide reveals predominantly an intergranular fracture mode, the grain size and shape has a significant influence on the mechanical properties. The toughness of materials with platelet-like grains is about twice as high as for materials with equiaxed grains. Materials exhibiting elongated microstructures show also a higher bending strength after post-HIPing. © 1999 Kluwer Academic Publishers

## 1. Introduction

Silicon carbide crystallizes in a cubic modification ( $\beta$ -SiC) and high number of different polytypes with a hexagonal or rhombohedral crystal symmetry ( $\alpha$ -SiC). The cubic modification is metastable and transforms at higher temperatures into one of the  $\alpha$ -polytypes [1]. The transformation temperature depends on impurities and is relatively sluggish due to the small difference in free energy of 2 kJ/mol between both modifications [2]. Jepps and Page [3] showed that the 6H  $\alpha$ -SiC polytype is stabilized in the presence of boron, whereas the 4H-polytype is stabilized by aluminum. The incorporation of aluminium and nitrogen leads to the formation of the 2H-polytype. The donor dopants N and P increase the stability of the cubic modification ( $\beta$ -SiC) so that a reverse transformation from  $\alpha$ -(6H) to  $\beta$ -SiC could be obtained under nitrogen overpressure [4, 5].

Silicon carbide ceramics (SSiC) are usually densified by pressureless sintering with carbon and boron or aluminium as sintering additives in a temperature range between 2100 °C and 2300 °C as described by Prochazka [6]. Carbon is necessary for the removal of

silica from the surface of the SiC-particles in order to decrease the surface energy. The diffusion of boron into the SiC-crystal structure increases the vacancy concentration and diffusivity. The resulting microstructure depends on the crystallographic modification of the SiC starting powder and varies between fine-grained equiaxed in case of  $\alpha$ -SiC and coarse-grained platelet-like grains when abnormal grain growth takes place during phase transformation from  $\beta$ -SiC into  $\alpha$ -SiC [7]. Nevertheless, the microstructure has only a minor influence on the mechanical properties due to the strong interfacial bonding between the grains: boron-doped SiC ceramics reveal a transgranular fracture mode and a fracture toughness between 2.5 and 4 MPa $\sqrt{m}$ . The advantages of this type of SiC ceramics are the excellent creep and oxidation resistances.

F. F. Lange [8] showed in 1975 an alternative densification method for silicon carbide ceramics. He obtained dense SiC ceramics by using hot-pressing at 1950 °C with  $Al_2O_3$  as a sintering additive. The  $Al_2O_3$  reacts with the  $SiO_2$ , present at the SiC-particle surfaces, and forms an eutectic melt well below the melting point

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of  $\text{Al}_2\text{O}_3$  so that densification will be achieved by liquid phase sintering (LPS). Complete densification by pressureless sintering could be obtained at sintering temperatures of  $1950^\circ\text{C}$  by using a mixture of  $\text{Y}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$  [9–12]. Böcker *et al.* [13] investigated the densification of LPS-SiC with  $\text{Y}_2\text{O}_3$  and AlN at sintering temperatures of  $1900^\circ\text{C}$  and observed that Al as well as N is dissolved in the SiC grains. Similar observations had been reported by Kleebe and Sigl [14] on plasma-etched samples.

After densification, the additives form an amorphous or partially crystalline grain boundary phase in triple junctions and two grain boundaries. The morphology of the SiC grains depends on the sintering conditions and the crystallographic modification of the initial SiC powder:  $\alpha$ -polytypes will not transform during sintering and give an equiaxed homogeneous microstructure, whereas  $\beta$ -SiC could transform into  $\alpha$ -SiC. The phase transformation is combined with the growth of platelet-like grains and offers the opportunity of an in-situ microstructural reinforcement similar to silicon nitride ceramics [15, 16]. The fracture toughness as well as the strength of LPS-SiC is higher in comparison to boron-doped SSiC due to a weaker interfacial bonding of the silicon carbide grains which causes a more intergranular fracture mode. Padture [15] reports a fracture toughness of  $7\text{ MPa}\sqrt{\text{m}}$  and Böcker and Hamminger [17] a strength of  $500\text{--}600\text{ MPa}$  which can be further improved to  $1000\text{ MPa}$  by post-HIPing. Since phase transformation plays an important role for the microstructural development and the resulting properties, detailed investigations of the transformation kinetics are necessary. The present paper analyses the dominant parameters which control the phase transformation and the development of the grain morphology and gives a relationship between microstructure and mechanical properties.

## 2. Experimental procedure

Different SiC materials were used for this work: 6H type  $\alpha$ -SiC (UF-15, Lonza GmbH, Waldshut, Germany) and  $\beta$ -SiC (Carbogran, Ividen, Japan); the sintering additives were  $\text{Y}_2\text{O}_3$  (grade fine, H.C. Starck GmbH, Goslar, Germany) and AlN (grade fine, H.C. Starck GmbH, Goslar, Germany). The starting powders were homogenized by attrition milling for 4 hours with  $\text{Si}_3\text{N}_4$  milling balls and isopropanol as milling media, subsequently dried in a rotation evaporator and sieved. Powder batches were consolidated by cold isostatic pressing at a pressure of  $630\text{ MPa}$  to cylindrical green compacts with a height and diameter of  $10\text{ mm}$ , respectively. The rectangular green compacts for measurements of the mechanical properties had a dimension of  $10\cdot 30\cdot 60\text{ mm}^3$ . Sintering experiments were performed in a graphite resistance furnace (Thermal Technology, USA) in a temperature range from  $1925^\circ\text{C}$  to  $2000^\circ\text{C}$  under various atmospheres and pressures. All specimens were sintered in a BN-crucible without a powder bed. The weight loss depends on the sintering conditions and varies between 2 and 5 wt %. The final density was determined by us-

ing Archimedes' method with water as the immersion medium.

The sintered samples were cut in two halves. One half was powdered for X-ray measurements to analyse the crystallographic modification of SiC after sintering. The analysis was performed with  $\text{Cu}_{\text{K}\alpha}$ -radiation using a standard powder diffractometer (Siemens D 5000, Germany) in an angle range  $2\theta$  between  $32$  and  $39^\circ$ . The quantitative analysis is based on the ratio of the (101)-peak for the 6H-polytype and the (111)-peak for the  $\beta$ -SiC phase [18]. The other halves of the cylindrical samples were ground and finally polished with  $1\ \mu\text{m}$  diamond paste for microstructural analysis. The surfaces were etched for 3 min by plasma-etching with a gas mixture of  $\text{CF}_4$  and  $\text{O}_2$  (ratio 1 : 1) at a total pressure of  $0.1\text{ mbar}$ .

Fracture toughness and hardness were determined by the indentation method with a load of  $10\text{ kg}$ . The corresponding  $K_{\text{Ic}}$ -values were calculated by using the equation of Anstis and Chantikul [19, 20]. Room temperature strength was measured by 4-point bending strength with an inner and outer span of  $40$  and  $20\text{ mm}$ , respectively, and sample dimensions of  $3\cdot 4\cdot 50\text{ mm}^3$ . Some of the sintered samples were additionally post-HIPed with a pressure of  $150\text{ MPa}$  at  $1900^\circ\text{C}$  in  $\text{N}_2$ -atmosphere.

## 3. Results and discussion

### 3.1. Influence of the sintering atmosphere and additive composition on the phase transformation kinetics

The phase transformation kinetics was studied with a powder mixture having a  $\alpha/\beta$ -SiC ratio of 1 : 9; the additive content was  $10.34\text{ wt}\%$   $\text{Y}_2\text{O}_3$  and  $2.95\text{ wt}\%$  AlN. Fig. 1 shows the  $\alpha$ -SiC content after pressureless sintering at  $1925^\circ\text{C}$  in nitrogen atmosphere as a function of sintering time. The initial  $\alpha$ -SiC content of 10% increases with increasing annealing time, but the transformation rate decreases. Even after 14 hours, phase transformation was still incomplete. The transformation kinetics could be described by a nucleation and grain growth process according to the equation

$$y(t) = a + (b - a) \cdot (1 - e^{-kt})$$

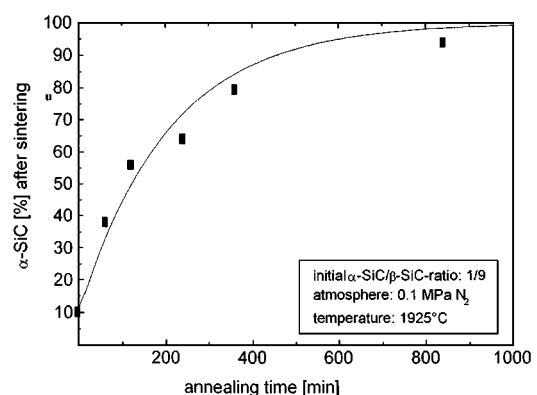


Figure 1 Phase transformation kinetics for a powder mixture with an initial  $\alpha/\beta$ -ratio of 1 : 9 at  $1925^\circ\text{C}$  and  $0.1\text{ MPa}$  nitrogen pressure.

which is a modified version of the equation derived by Johnson and Mehl [21] for grain growth processes.  $y$  gives the  $\alpha$ -content at time ( $t$ ),  $a$  and  $b$  are constants for the initial ( $a$ ) and final ( $b$ )  $\alpha$ -content; the constant  $k$  describes the transformation kinetics. The observed transformation behaviour can be explained by a steric hindrance of grain growth of  $\alpha$ -SiC grains. At the beginning of the annealing time,  $\alpha$ -SiC particles could grow without any impingement, resulting in high transformation rates. With increasing time, the growing grains start to touch each other due to a pronounced grain growth anisotropy with high growth rates of the prism planes and low growth rates of the basal planes. Since grain growth is hindered, the transformation rate decreases. A similar behaviour has been observed in the case of the phase transformation from  $\alpha$ - to  $\beta$ -Si<sub>3</sub>N<sub>4</sub> [22].

A further set of experiments were performed with powder mixtures containing the same amount and composition of additives, but different  $\alpha/\beta$ -ratios in order to analyse the influence of the initial  $\alpha$ -content. The specimens were again pressureless sintered for 30 min at 1925 °C in nitrogen atmosphere and subsequently annealed for 6 hours at the same temperature, but under 1.5 MPa Ar-pressure. Fig. 2 reveals a complete transformation for compositions with initial  $\beta$ -contents  $\leq 50\%$ ; powder mixtures with higher initial  $\beta$ -contents exhibit residual non-transformed  $\beta$ -SiC. The specimen which contains  $\beta$ -SiC as the only modification in the starting powder shows no transformation at all. Therefore it can be concluded that phase transformation requires the presence of  $\alpha$ -SiC nucleation sites. The homogeneous nucleation of  $\alpha$ -SiC or the heterogeneous nucleation on  $\beta$ -SiC could be neglected. Under this assumption it is easy to understand the strong influence of the initial  $\alpha$ -content on the transformation rate, since the  $\alpha$ -SiC content correlates directly with the number of nucleation sites. The results seem to be in contradiction with the observations of Lee [16] and Mulla and Krstic [23], who reported a phase transformation from  $\beta$ -SiC into  $\alpha$ -SiC (4H-type) in samples with pure  $\beta$ -SiC starting powder. However, they used additive compositions with a higher Al content. Indeed, the transformation strongly depends on the additive composition. Fig. 3 shows the X-ray diagram for pure  $\beta$ -SiC in comparison to diffraction pat-

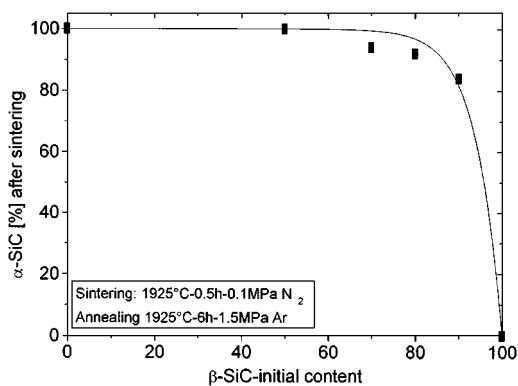


Figure 2  $\alpha$ -SiC content as a function of the initial  $\beta$ -content after 30 min sintering at 1925 °C at 0.1 MPa nitrogen pressure and a subsequent heat treatment for 6 hours at 1.5 MPa argon.

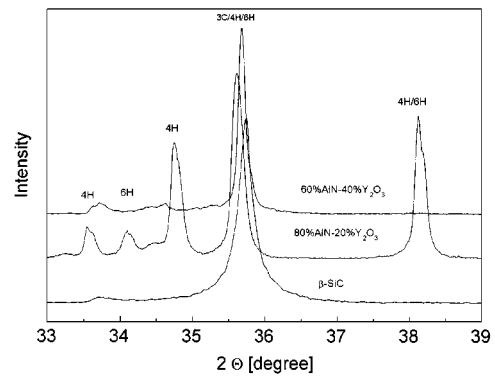


Figure 3 X-ray diffraction pattern of the  $\beta$ -SiC starting powder in comparison to the diffraction patterns of LPS-ceramics pressureless sintered at 1925 °C in Ar atmosphere with the additive system: 80 mol-% AlN/20 mol-% Y<sub>2</sub>O<sub>3</sub> and 60 mol-% AlN/40 mol-% Y<sub>2</sub>O<sub>3</sub>, respectively.

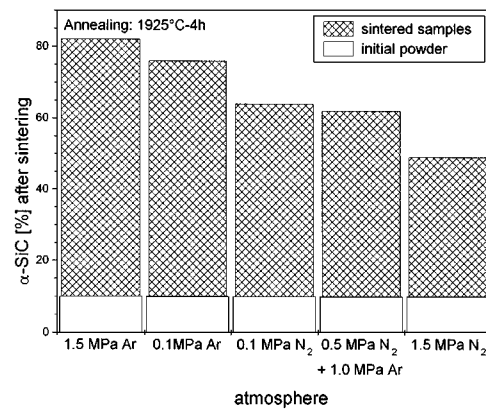


Figure 4 Influence of the sintering atmosphere and pressure on the final  $\alpha$ -SiC content for powder mixtures with an initial  $\beta$ -SiC content of 90%.

terns for two materials with different AlN/Y<sub>2</sub>O<sub>3</sub>-ratios sintered at 1925 °C for 3 hours in Ar atmosphere. The AlN-rich additive composition exhibits a phase transformation, but the composition with the lower AlN content did not transform. Some additional peaks indicate the presence of a crystalline grain boundary phase.

The influence of the sintering atmosphere was finally studied by using a powder with an  $\alpha/\beta$  ratio of 1 : 9 and a constant additive composition of 10.34 wt % Y<sub>2</sub>O<sub>3</sub> and 2.95 wt % AlN. Fig. 4 reveals the obtained  $\alpha$ -SiC content after sintering at 1925 °C for 4 hours in different atmospheres. The transformation in Ar atmosphere is faster than in a N<sub>2</sub> atmosphere. Furthermore it could be seen that the transformation accelerates with an increase in Ar-pressure, but an increase of the N<sub>2</sub> pressure has the opposite effect. A reverse transformation from  $\alpha$ - to  $\beta$ -SiC under high nitrogen pressure, as reported by Jepps and Page [5], could not be confirmed. The influence of the sintering atmosphere in the case of the present results is attributed to the presence of a liquid phase during sintering. An increasing Ar-pressure leads to an increase in the SiC-dissolution because of the higher solution pressure of SiC-particles so that the transformation rate increases. Under the assumption of a preferred nitrogen solubility in the liquid phase, the SiC solubility will decrease compared to an Ar-atmosphere. An increasing sintering pressure reinforces this effect and the transformation rate will decrease.

### 3.2. Influence of phase transformation on microstructural development

The possibilities to tailor the final microstructure of LPS-SiC by control of the  $\alpha/\beta$ -ratio of the starting powder is shown in Fig. 5a–d. The specimens were pressureless sintered in  $N_2$ -atmosphere at 1925 °C for 1 and 14 hours, respectively. The plasma-etched surfaces of Fig. 5a indicate a relatively homogeneous microstructure with nearly equiaxed grains for samples prepared from a powder containing 100%  $\alpha$ -phase. The grain size increases from approximately 0.6  $\mu\text{m}$  to 1.5  $\mu\text{m}$  during subsequent isothermal sintering for 13 hours, but the grain morphology is still nearly equiaxed (Fig. 5b). Specimens with an initial  $\alpha/\beta$ -ratio of 1:9 reveal a similar microstructure with equiaxed grains and a mean grain size of approximately 0.7  $\mu\text{m}$  after 1 hour isothermal sintering, Fig. 5c. However, after 14 hours isothermal sintering the grain morphology changes: the grains become more elongated and the mean grain size increases in comparison to the material prepared from pure  $\alpha$ -powder, Fig. 5d. All microstructures reveal grains with a typical core shell structure, visible after plasma-etching. The different etching rate of core and shell is attributed to the solution of the sintering additive aluminium and probably nitrogen in the SiC crystal structure. The phenomena has already been described by Kleebe and Sigl [14]. The deeper etched core represents the initial grain without Al and N, whereas the shell grows via a solution reprecipitation process in the presence of an Al and N-rich phase during sintering and subsequent grain coarsening. Fig. 6 shows the corresponding X-ray analysis data of the four samples for an angle range from 33 to 39° ( $2\theta$ ). Specimens prepared from pure  $\alpha$ -powder (6H-type) show no change in the peak intensity. After 14 hours of isothermal sintering the crystallization of a grain boundary phase ( $\text{AlYc}_{0.5}$ -phase) could be observed. The  $\alpha/\beta$ -mixture shows an increasing phase transformation from  $\beta$ -SiC to  $\alpha$ -SiC with sintering time as indicated by the increasing intensity for the 6H/4H-peaks. A crystallization of the grain boundary could also be detected.

From the results it could be concluded that a significant change of the grain morphology could be obtained in the presence of a phase transformation, whereas pure  $\alpha$ -SiC or  $\beta$ -SiC did not transform with the standard additive system; the grains remain equiaxed even for long sintering times. Since transformation is relatively slow, the formation of elongated grains with a platelet-like morphology requires long sintering times. A comparison of the microstructures of a partially transformed specimen (Fig. 6a) and the completely transformed one reveals that most of the phase transformation occurs after complete densification. In section 3.1 it has already been pointed out that the density of nuclei will have a strong influence on the phase transformation kinetics and therefore on the microstructure. In case of a low  $\alpha$ -SiC nuclei density only a few particles are able to grow. These grains should be able to grow in the “ $\beta$ -SiC matrix” for long sintering times with only a minor steric hindrance, resulting in an increasing grain aspect ratio. In order to prove this argument, compositions with an initial  $\alpha$ -content of 1, 10 and 100% have been densified at 2000 °C for 4 hours under atmospheric

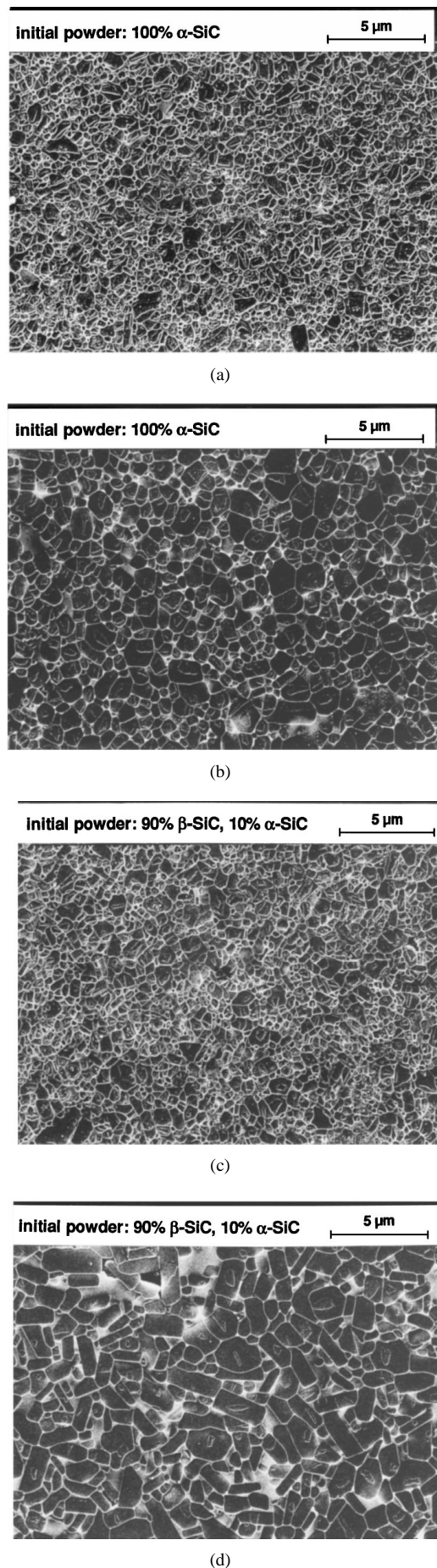


Figure 5 Plasma-etched SEM-micrographs of LPS-samples with various initial  $\alpha/\beta$ -SiC ratios after pressureless sintering in nitrogen atmosphere at 1925 °C for 1 and 14 hours: (a) 100 pure  $\alpha$ -SiC (6H-type)/1 h, (b) 100%  $\alpha$ -SiC/14 hours, (c) 10%  $\alpha$ -SiC and 90%  $\beta$ -SiC/1 h, (d) 10%  $\alpha$ -SiC and 90%  $\beta$ -SiC/14 hours.

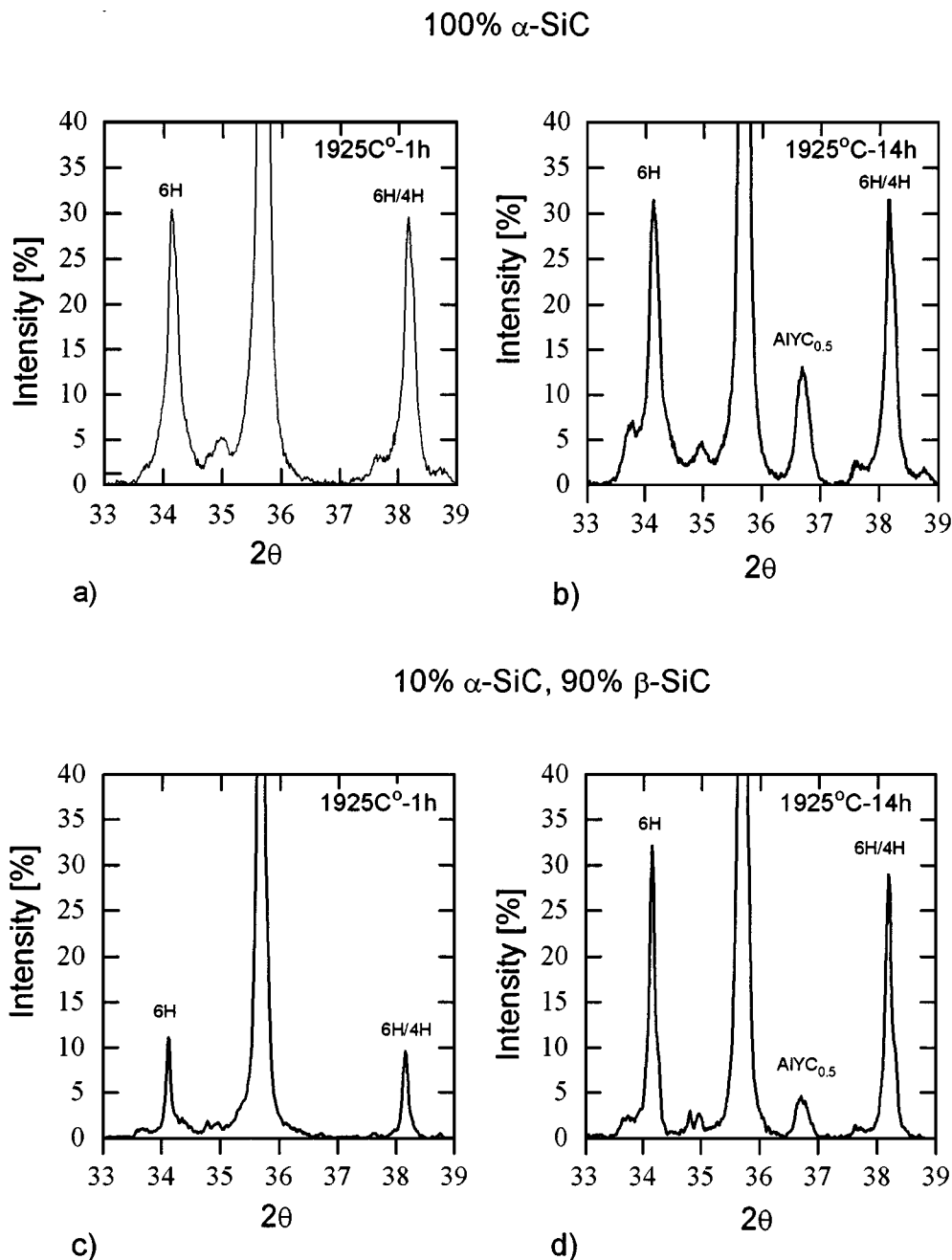
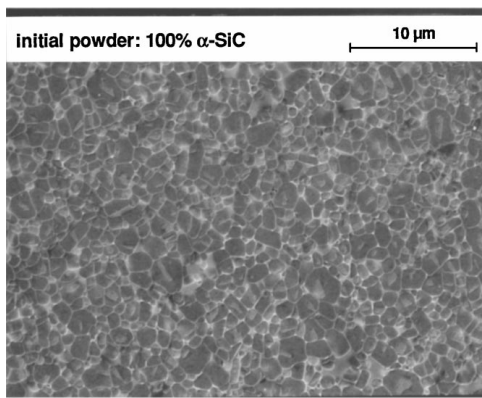


Figure 6 XRD-patterns of samples with various  $\alpha/\beta$ -SiC ratios of the starting powder after pressureless sintering in nitrogen atmosphere at 1925 °C: (a) 100%  $\alpha$ -SiC/1 h, (b) 100%  $\alpha$ -SiC/14 hours, (c) 10%  $\alpha$ -SiC/1 h, (d) 10%  $\alpha$ -SiC/14 hours.

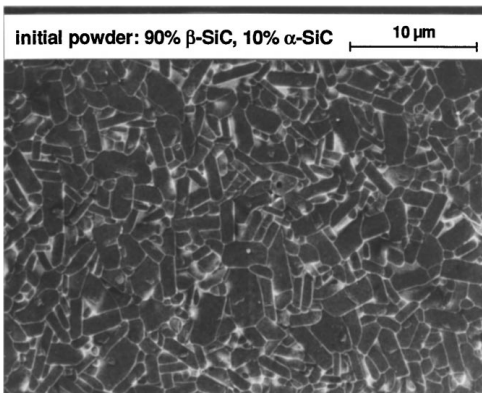
pressure and subsequently for 30 min at 1.5 MPa  $N_2$ -pressure. The obtained microstructures of the three compositions, which are all completely transformed after sintering, are shown in Fig. 7. The sample prepared with pure  $\alpha$ -SiC reveals a globular microstructure (Fig. 7a), while compositions with mainly  $\beta$ -SiC as starting powder consist of elongated grains. It is obvious from Fig. 7b and c, that the grain aspect ratio increases with an increasing initial  $\beta$ -SiC content. The opportunity to control the microstructural evolution by the number and size of  $\alpha$ -grains in a starting powder is similar to  $Si_3N_4$ -ceramics [24]. Nevertheless, the grain shape differs between both materials:  $Si_3N_4$  crystals reveal a needle-like morphology due to a high growth rate of the basal planes in comparison to the prism planes, whereas in case of SiC the prism planes grow faster and the grain morphology becomes platelet-like.

### 3.3. Mechanical properties

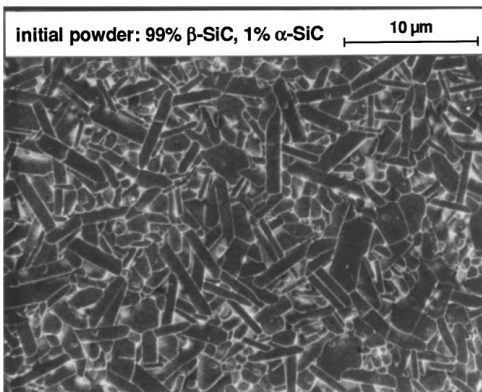
The plasma-etched microstructures show SiC-grains, which are well separated by an amorphous or partially crystalline grain boundary phase. The  $Y_2O_3$ -rich secondary phase is assumed to be weaker than the SiC-grains so that an intergranular fracture mode could be observed. The crack path and the energy required for crack propagation will therefore be strongly influenced by the size and morphology of the grains. Fig. 8 shows the influence of the microstructure on the fracture toughness for compositions sintered at 1925 °C in  $N_2$ -atmosphere with an initial  $\alpha$ -content of 100% and 10%, respectively. The  $K_{Ic}$  for the material prepared from pure  $\alpha$ -powder decreases from 4.6 to 3.6  $MPa\sqrt{m}$  with increasing isothermal sintering, whereas the one for the  $\beta$ -rich material shows the opposite behaviour. Fig. 9 reveals the fracture toughness as a function of the



(a)



(b)



(c)

Figure 7 Plasma-etched SEM-micrographs of LPS-samples with different initial  $\alpha/\beta$ -SiC ratios after pressureless sintering at 2000 °C for 4 h and a subsequent heat treating for 30 mins at a pressure of 1.5 MPa nitrogen: (a) 100%  $\alpha$ -SiC, (b) 10%  $\alpha$ -SiC (c) 1%  $\alpha$ -SiC.

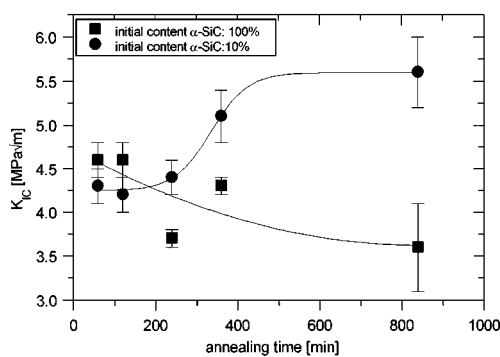


Figure 8 Indentation fracture toughness as a function of sintering time at 1925 °C under atmospheric nitrogen pressure for samples with an initial  $\alpha$ -SiC content of 10 and 100%.

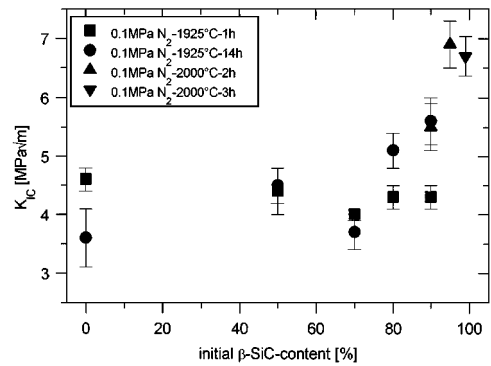


Figure 9 Dependency of the indentation fracture toughness on the initial  $\beta$ -SiC content.

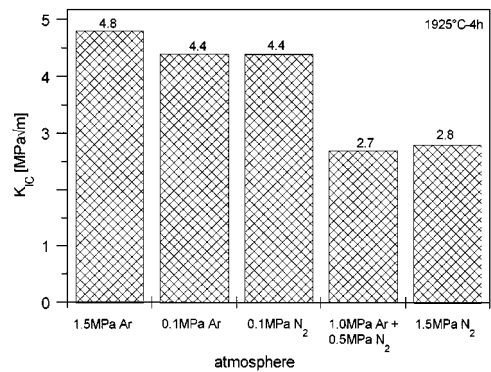


Figure 10 Indentation fracture toughness of LPS-ceramics with initially 90%  $\beta$ -SiC after sintering at 1925 °C for 4 hours under various atmospheres and pressures.

initial  $\beta$ -SiC content. The  $K_{IC}$  of the samples sintered at 1925 °C for 1 hour is independent of the initial  $\alpha/\beta$ -ratio due to the small transformation rate. However, samples sintered at higher temperatures or longer sintering times are completely transformed into  $\alpha$ -SiC and reveal a strong increase in fracture toughness from 3.7 to 6.7 MPa $\sqrt{m}$ . The toughness increase is attributed to the development of elongated grains during phase transformation, as already demonstrated in the previous section. Grains with high aspect ratios offer the possibility to deflect the crack tip or to bridge crack wakes. The microstructural reinforcement by the growth of grains with a platelet-like morphology and the resulting toughening mechanisms are comparable with the “in-situ reinforcement” of  $Si_3N_4$ -microstructures by needle-like grains [25].

The results of the transformation kinetics revealed a slower transformation when sintering is performed in nitrogen atmosphere. Therefore fracture toughness should decrease with increasing nitrogen pressure, as is shown in Fig. 10 for samples containing initially 90%  $\beta$ -SiC. The highest value could be obtained with 1.5 MPa Ar-pressure. However, samples sintered under 0.1 MPa Ar- and  $N_2$ -pressure reveal the same  $K_{IC}$ , whereas it decreases for higher  $N_2$ -pressures. A comparison of Figs 4 and 10 indicates that the transformation rate could be related to the  $K_{IC}$ -values. Nevertheless, the toughness for samples sintered in a  $N_2$ -atmosphere is higher than expected from phase transformation. This can be attributed to a change in the chemical composition of the intergranular phase

TABLE I Bending strength for LPS-ceramics with different initial  $\alpha/\beta$ -ratios. (PHIP) denotes post-HIPing at 1900 °C for 30 min at 150 MPa Ar

Initial $\alpha/\beta$ -SiC ratio	Sintering program	Strength [MPa]
100% $\alpha$	1925 °C-4 h 0.1 MPa N <sub>2</sub> -0.5 h 1.5 MPa Ar	482 ± 87
$\alpha/\beta = 1/9$	1925 °C-4 h 0.1 MPa N <sub>2</sub> -0.5 h 1.5 MPa Ar	532 ± 56
100% $\alpha$	1925 °C-4 h 0.1 MPa N <sub>2</sub> -0.5 h 1.5 MPa Ar (PHIP)	587 ± 170
$\alpha/\beta = 1/9$	1925 °C-4 h 0.1 MPa N <sub>2</sub> -0.5 h 1.5 MPa Ar (PHIP)	592 ± 114
$\alpha/\beta = 5/95$	2000 °C-2 h 0.1 MPa N <sub>2</sub> -0.5 h 1.5 MPa Ar (PHIP)	585 ± 57
$\alpha/\beta = 1/99$	2000 °C-3 h 0.1 MPa N <sub>2</sub> -0.5 h 1.5 MPa Ar (PHIP)	614 ± 105

during sintering in Ar- or N<sub>2</sub>-atmosphere. It is assumed that nitrogen could be dissolved in the liquid phase and weaken the interfacial strength between SiC-grains compared to Ar-sintered materials. Weaker interfaces can activate more toughening mechanisms at the crack tip and crack wake. The strong effect of the chemical composition of the intergranular phase on the toughness has also been reported from Si<sub>3</sub>N<sub>4</sub>-ceramics with different Y<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub>-ratios and nitrogen contents in the liquid phase [26].

The bending strength was measured for four compositions to study the influence of the initial  $\alpha/\beta$ -ratio and the effect of post-HIPing. The results are summarized in Table I together with the starting composition and sintering conditions. The mean strength of samples with a fine-grained microstructure made from pure  $\alpha$ -powder was 482 MPa. This strength could be increased to 532 MPa when a powder with an  $\alpha/\beta$ -ratio of 1 : 9 is used under the same sintering conditions. An improvement of the strength to 587 and 592 MPa, respectively, could be obtained by post-HIPing for 30 min at 1900 °C and 150 MPa Ar-pressure. The microstructure with the most elongated grains and the highest fracture toughness also reveals the highest mean strength of 614 MPa, with individual data varying from 758 to 498 MPa. The results show only a tendency due to the limited number of only 6 tested samples for each composition. Nevertheless, the data reflect an increasing strength with an increasing  $\alpha/\beta$ -ratio of the starting powder in combination with a phase transformation. Fractographic investigations of the fracture surfaces by SEM show residual pores as the fracture origin for all samples. Therefore it is assumed that the strength could be significantly enhanced by an optimization of the processing conditions and sintering parameters.

#### 4. Conclusion

A detailed analysis of the phase transformation kinetics from  $\beta$ - to  $\alpha$ -SiC and the corresponding microstructural development of liquid phase sintered SiC is reported. Experiments with different  $\alpha/\beta$ -ratios of the starting powder indicate that the transformation rate is controlled by the number of  $\alpha$ -SiC nucleation sites and the additive composition, beside the usual parameters of sintering time and temperature. The transformation kinetics is relatively slow so that samples with an initial  $\beta$ -content of 90% require sintering times of >14 hours at 1925 °C for complete transformation. A further reduction of the transformation rate is found in the presence of a nitrogen atmosphere. The phase transformation could be used to control the microstructure of LPS-SiC

ceramics due to the anisotropic grain growth behaviour of SiC. In the absence of a phase transformation (pure  $\alpha$ -powder) a fine-grained equiaxed microstructure develops, whereas the aspect ratio of the grains increases with a decreasing initial  $\alpha$ -content. The fracture toughness of samples containing in-situ grown high aspect ratio SiC platelets is about twice that of samples with equiaxed grains. Bending strength measurements indicate a significantly higher strength in comparison to conventional sintered silicon carbide with boron and carbon as sintering additives. The strong influence of the microstructure on the mechanical properties is attributed to the intergranular fracture mode.

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