Mechanical Properties of SiC-AlN Ceramic Composites

Jing-Feng LI and Ryuzo WATANABE

Department of Materials Processing
Faculty of Engineering
Tohoku University
Sendai 980 , JAPAN

ABSTRACT

SiC-AlN particulate composites with compositions ranging from 5 to 100mol% AlN were fabricated by hot-isostatic pressing of powder mixtures of SiC and AlN. The microstructure consists of SiC-rich and AlN-rich phases with varying compositions. Mechanical properties such as flexural strength, fracture toughness and Vickers hardness were determined as a function of composition. Remarkable strengthening and toughening effects were achieved in the SiC-AlN composites, compared with conventional monolithic SiC.Mechanical properties can be controlled over a wide range by controlling the composition and microstructure of the composites.

1) INTRODUCTION

It has been reported that solid solution is formed in the SiC-AlN system over a wide composition range[1-6]. Since material properties are widely varied by solid solutioning, SiC-AlN alloys are expected to open a new field of practical application of SiC and AlN ceramics. On the other hand, it has been known that a ceramic composite with a heterogeneous microstructure always shows higher toughness than homogeneous monolithic ceramics due to the interaction between cracks and dispersion particulates[7,8]. In some cases, the SiC-AlN mixed composites are unexpectedly obtained, when solid solutioning is insufficient[3]. It is necessary indeed to investigate the mechanical properties of SiC-AlN particulate composites from the view point of the applicability of engineering ceramics. The SiC-AlN composite is easier to fabricate by hot-isostatic pressing of the powder mixtures of SiC and AlN at lower temperatures than hot-pressing.

In the present study, SiC-AlN particulate composites were prepared by hot-isostatic pressing of the powder mixture of SiC and AlN. The compositional dependence of mechanical properties was investigated and discussed.

2)EXPERIMENTAL PROCEDURE

2.1 Sample preparation

Commercially available SiC powder (Showa Denko K.K. Company, Japan) and AlN powder (Toyo Aluminum Company, Japan) were used as raw materials. The specifications of the powders are listed in Table.1. Powder mixtures with the compositions ranging from 2 to 90mol% AlN were prepared by ball-milling in an alcohol solution and drying at 400K for 48h. Green compacts (10mm diameter by about 30mm height) were formed by uniaxial pressing in a steel die at 100MPa and subsequent cold-isostatic pressing at 200MPa. The green compacts were coated with BN powders, and encapsulated in a Vycor glass tube under a vacuum of 0.01Pa.

The glass-encapsulated specimens were hot-isostatically pressed at 200 MPa in a temperature range from 2073 to 2223K for 2h. The heating and pressurizing schedules are presented in Fig.1.

The sintered density was measured by the water displacement method. Fully dense specimens obtained by hot-isostatic pressing at 2123K were used for the measurement of mechanical properties.

2.2 XRD and STEM-EDX analysis

X-ray diffraction (XRD) analysis was done on the polished surface of the sintered compact. Ni-filtered CuKα radiation was used. Thin film specimens with a diameter of about 3mm were prepared by diamond-polishing and argon-ion thinning for microstructure observation and composition microanalysis within grains by a scanning transmission electron microscope (STEM) equipped with an energy-dispersive X-ray spectrometer (EDX).

2.3 Measurement of mechanical properties

Flexural strength was measured by 4-point bending test.The specimens (length=23mm, width=2.5mm,thickness=1.5mm) for bending test were cut from the sintered compacts using a diamond cutter. The tensile surface was polished with 15-. 6-. and 3 μm diamond paste to mirror-like surface. Bending test was conducted at a crosshead speed of 0.05 mm/min. The spans of the upper and lower supporting pins were 10.0 and 20.0mm, respectively.

Microhardness and fracture toughness were measured using the fractured bending-test specimens. Vickers indentations were made on the mirror-like surface using a 4.9N load for the measurement of microhardness, and a 49N for fracture toughness, respectively. The fracture toughness value was calculated from the equation proposed by K.Niihara et al.[9].

3) RESULTS AND DISCUSSION

Fig.2 shows the relationship between relative densities of sintered compacts and sintering temperatures. As shown in the figure, no full-dense SiC compacts were obtained at the temperatures up to 2223K, while a mixture of SiC and AlN was densified to theoretical density even at the lower temperatures. The sinterability was increased with increase in AlN content, showing a sintering enhancement by the AlN addition to SiC. Dense SiC-AlN composites with compositions of more than 5mol% AlN were obtained by hot-isostatic pressing at 2123K without other additives. For a comparison, dense SiC compact was prepared by hot-isostatic pressing at 2173K with additions of 0.5mass% B_4C.

Even though SiC and AlN can form a solid solution over a wide composition range, it has been reported that low diffusion coefficients prevent the formation of homogeneous solid solution by sintering the powders mixture of SiC and AlN[1,3]. As shown in Fig.3 , the XRD profile indicates that SiC and AlN exist in the sintered compact. A more detailed

analysis of chemical composition was conducted by STEM-EDX. Fig.4 shows the microstructure of SiC-25mol%AlN composite together with the data of EDX compositional analysis. As can be seen from this figure, all composites consist of AlN-rich larger grains and SiC-rich finer grains, and the grains with intermediate composition are also formed between the SiC- and AlN-rich grains. A compositional gradient seems to exist within the grains, as has been suggested by Ruh and Zangvil[3]. While, from the EDX results , it is clear that the interdiffusion between SiC and AlN particles occurred in the sintering process, which is likely to enhance the sintering densification of the compacts.

Fig.5 shows flexural strength as a function of composition. Monolithic SiC (with 0.5mass% B_4C additions) has an average strength of 500MPa, in good agreement with reported values. The strength of the composite showed the maximum at 25mol% AlN with an average value of 640MPa, and decreased to a minimum value of about 400MPa at the composition of 90mol%AlN, The compositional dependence of strength was found to be significantly affected by mixing uniformity. The strengthening effect could not be achieved in the composite with an inhomogeneous microstructure. The similar behavior was reported for the composites fabricated by hot-pressing[3]. The composite with a maximum strength over 700MPa could be obtained by more homogeneous mixing of the starting powders.

Fig.6 shows the compositional dependence of fracture toughness determined by the indentation microfracture method. The additions of AlN particulate into SiC matrix effectively increased the fracture toughness. The composite with the composition of 50mol% AlN has the maximum value nearly two times that of monolithic SiC. In the AlN rich side, the same toughening effect was achieved by the mixing of SiC. Fig.7 shows indentation cracks observed by a scanning electron microscopy (SEM). As seen in Fig.7 . the crack path in the monolithic SiC is very smooth, while those in the present composites are much more complicated. In the composites, it was observed that the cracks propagation was accompanied by crack deflection, crack branching and

particulate bridging. In the monolithic AlN, since the crack dominantly propagated intergranularly , the indentation crack path was jagged. The more complicated path the indentation crack has , the larger the crack propagation resistance is. As a result, the fracture toughness of the material is increased.

In composite materials, the mismatch of the coefficients of thermal expansion (CTE) and elastic moduli between a matrix and a dispersive particulate results in a generation of residual stress in the particulate and surrounding matrix , when the material is cooled from the fabricating temperature[7]. The magnitude of the residual stresses can be calculated from the coefficients of thermal expansion, the elastic moduli and Poisson's ratios, using formula proposed by Wei and Becher[7]. In the present work, CTEs and Young's moduli for SiC and AlN and their composites were measured. The measured CTE values of SiC and AlN are 4.8 and $5.7\times10^{-6}K^{-1}$,respectively. Hence, on the SiC rich side, the residual stress as shown in Fig.8(a) were generated. In this case, a crack moving in the plane of the AlN particulate will deflected out of the plane and oriented normal to the radial tensile stress axis. The crack deflection was actually observed in the SiC-25mol%AlN composite. On the contrary , on the AlN rich side, the opposite residual stresses were generated in the AlN matrix around the SiC particulate. In this case, as shown in Fig.8(b), since the opening of crack are suppressed by the radial compressive stresses, the mechanism such as the particulate bridging will result in the toughening effect. Actually in the composites with higher AlN contents,particulate bridging was often observed as shown in Fig.7. In addition, crack deflection may also occur due to the circumferential tensile stresses as shown in Fig.8(c).

In the SiC-AlN system, since interdiffusion occurs between the SiC and AlN particles, the interface between matrix and dispersive particulate is considered to be strongly bonded. The strong bonding is likely to contribute to the improvement of flexural strength on the SiC rich side. The residual stresses are also considered to be relaxed to some extent due to the interdiffusion. In addition, toughening effect due to solid

solutioning between SiC and AlN should also be taken into account.

The compositional dependence of microhardness was also measured. SiC has a high hardness (about 29.8GPa) of 2.4 times that of AlN (12.5GPa). The hardness varies almost linearly with the composition.

4)CONCLUSION

The SiC-AlN particulate composites were prepared by hot-isostatic pressing of powder mixtures of SiC and AlN at rather low temperature of 2123K. The interdiffusion occurred during sintering, and the densification of SiC was enhanced by a small amount of AlN additions. The obtained microstructures consist of SiC- and AlN-rich solid solutions with varying compositions.

The compositional dependence of the mechanical properties such as flexural strength, fracture toughness and microhardness were investigated for the fully dense composites. It has been found that strengthening can be achieved in the composites, particularly the strength was increased to 640MPa from 500MPa (for monolithic SiC) at the composition of SiC-25mol%AlN. The compositional dependence of fracture toughness showed the maximum nearly two times that of SiC at about 50mol% AlN. It has been emphasized that ceramic composites from powder mixtures will give a availability of controlled mechanical properties.

REFERENCES

1. I.B.Cutler, P.D.Miller, W.Rafaniello, D.P.Thompson, and H.K.Jack, "New Materials in the Si-C-Al-O-N and Related System," Nature (London), 275,434-35 (1978).
2. W.Rafaniello, K.Cho, and A.V.Virkar, "Fabrication and Charaterization of SiC-AlN Alloys," J. Mater.Sci., 16, No.12, 3479-88 (1981).
3. Ruh and A.Zangvil, "Composition and Properties of Hot-Pressed SiC-AlN Solid Solutions," J. Am. Ceram. Soc., 65, No.5, 260-65 (1982).
4. M.M.Dobson, in "Silicon Carbide Alloy," Research Reports in Materials Science, Series One, edited by P.E.Evans, (Parthenon, England, 1986).
5. J-F Li, A.Kawasaki, and R.Watanabe, "Synthesis of SiC-AlN Ceramic

Alloys by Reaction Sintering and its HIP-densification," J. Japan Soc. Powder and Powder Met., 36, No.6, 740-45 (1989).

6. C.L.Czekaj, M.L.J.Hackney, W.J.Hurley, Jr., Leonard V. Interrante, and Gray A.Sigel, "Preparation of Silicon Carbide/Aluminum Nitride Ceramics Using Organometallic Precursors," J. Am. Ceram. Soc., 73, No.2, 352-57 (1990).
7. G.C.Wei and P.F.Becher, "Improvements in Mechanical Properties in SiC by the Addition of TiC Particles," J. Am. Ceram. Soc., 67, No.8, 571-74 (1983).
8. K.T.Faber and A.G.Evans, "Crack Deflection Processes-I. Theory," Acta Metall. 31,No.4, 565-76 (1983).
9. K.Niihara, R.Morena, and D.P.H.Hasselman, "Evaluation of K_{IC} of brittle Solids by the Indentation Method with Low Crack-to-Indent Ratios," J. Mater. Sci. Lett., 1, No.1, 13-16 (1982).

Table 1 The specification of the starting powders.

Powder	Commerical name (grade)	Phase	Particle size (μm)	Main impurity (mass%)				
				C	O	Al	Fe	Si
SiC	Shoden(A-1)	α(6H)	0.37	0.70	0.37	0.01	0.04	---
AlN	Toyalnite(F)	α	1.39	0.08	0.99	---	0.0054	0.0116

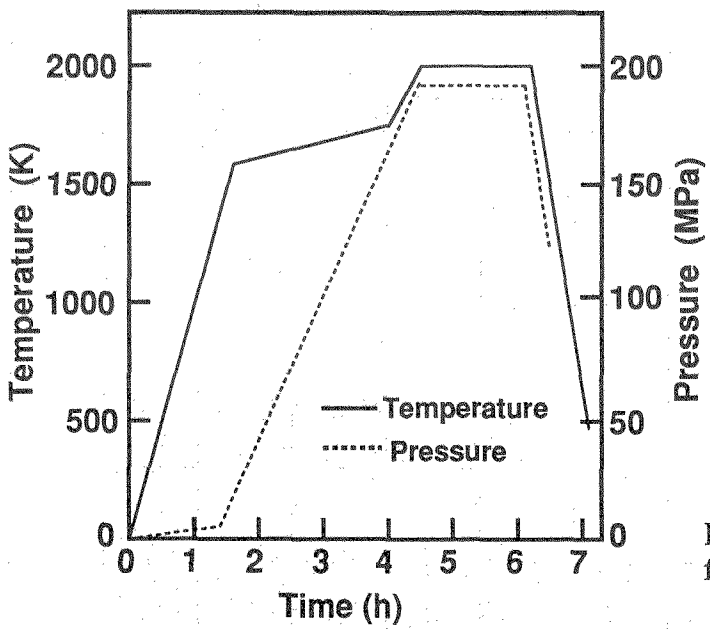

Fig.1 Schedule of operation for hot-isostatic pressing.

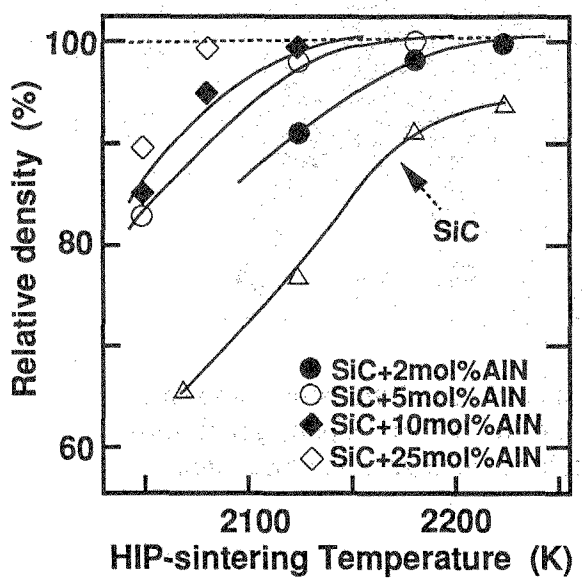

Fig.2 Relative density of sintered compact as a function of sintering temperature.

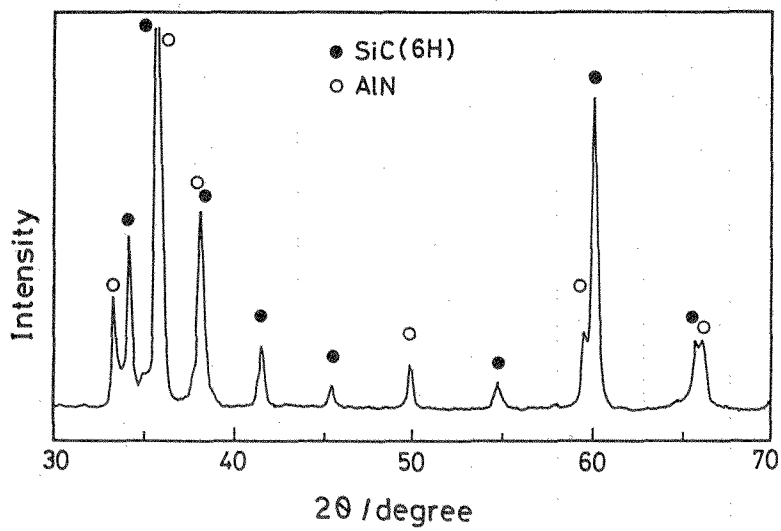

Fig.3 X-ray diffraction profile of SiC-25mol%AlN composites hot-isostatically pressed at 2123K for 2h.

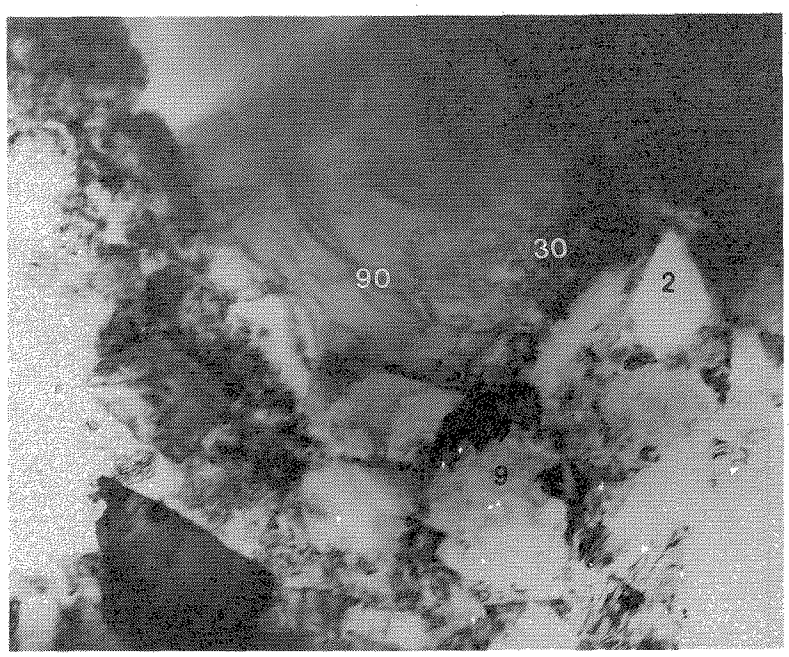

Fig.4 Transmission electron micrograph of the same composite investigated in Fig.3. The figures in the photograph show the AlN content by mole percentage in the local area.

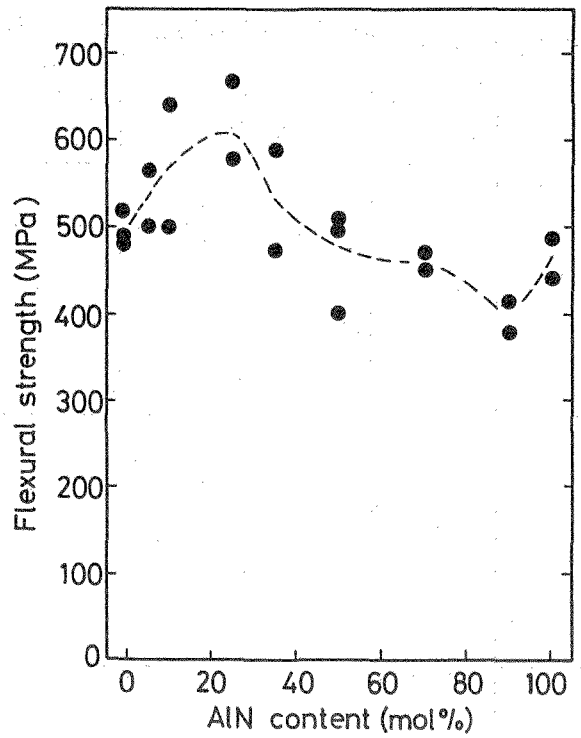

Fig.5 Flexural strength of SiC-AlN composites as a function of composition.

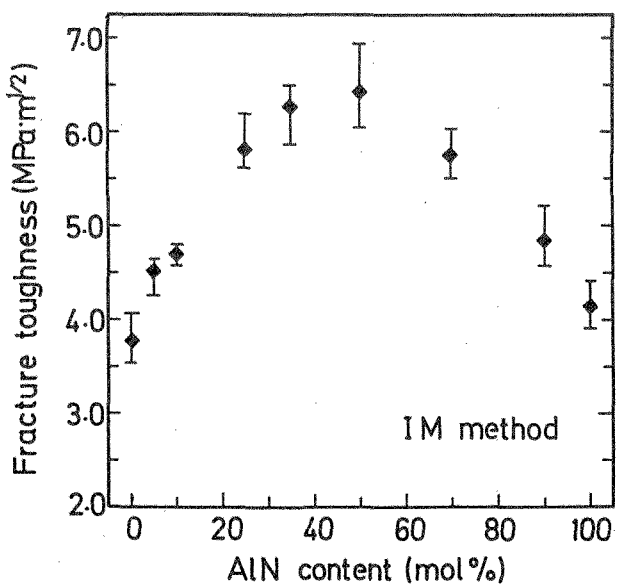

Fig.6 Fracture toughness as a function of AlN content , determined by the indentation method.

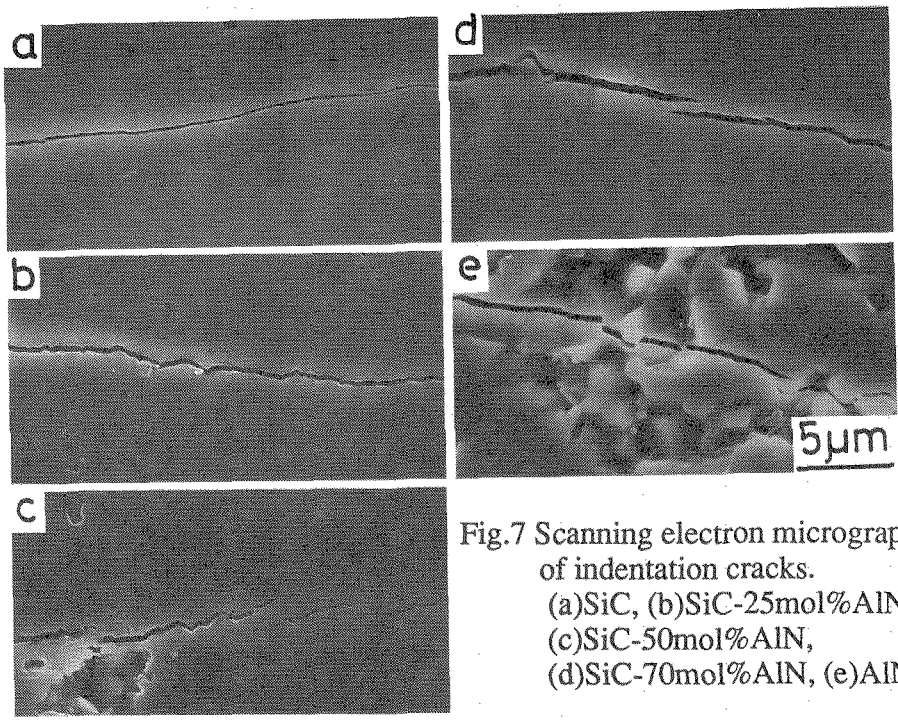

Fig.7 Scanning electron micrographs of indentation cracks.
(a)SiC, (b)SiC-25mol%AlN,
(c)SiC-50mol%AlN,
(d)SiC-70mol%AlN, (e)AlN.

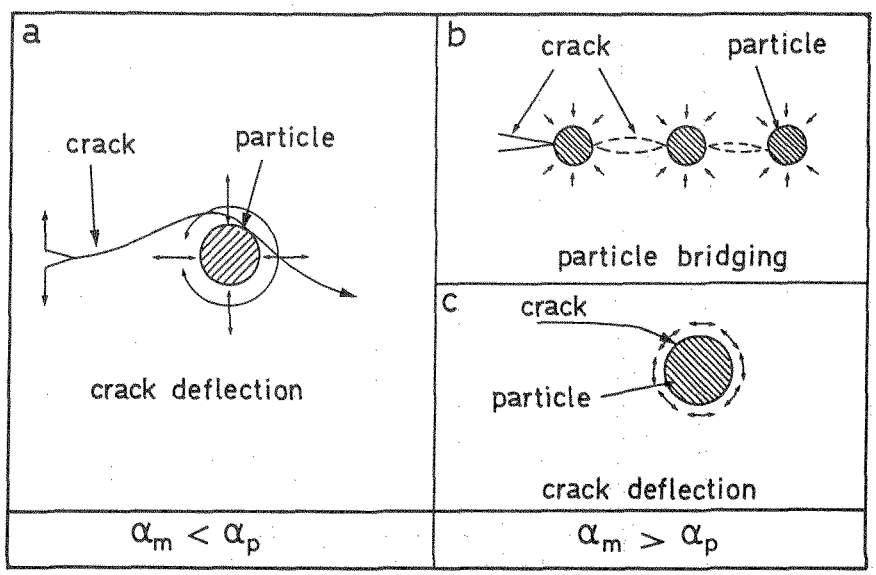

Fig.8 Schematic drawing of toughening mechanism in the SiC-AlN system.