

## In-situ Preparation of SiC Whiskers in $\text{Si}_3\text{N}_4$ Powder and the Properties of their Composites

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### ABSTRACT

In order to obtain thick SiC whiskers, in-situ prepared in  $\text{Si}_3\text{N}_4$  powder under pressure from  $\text{SiO}_2$  and C, NaCl was found to be needed as a co-existing material, giving space enough to admit the whisker growth in the matrix powder. For the in-situ preparation under normal pressure, iron powder having a particular size, was found to be effective to get thick whiskers having diameters of 1 - 2  $\mu\text{m}$ .

On the other hand, the bending strength and fracture toughness of the composite mentioned above, could not be improved. Trace amount of residual carbon was often observed as the fracture origin in scanning electron microscopy. Even after more complete elimination of the trace carbon, the properties were not improved, one reason of which was considered to be its inadequate homogeneity according to a comparison of the composite texture with that of a physically mixed one providing the highest strength and toughness. The other reasons may be the existence of coarse particles at the fracture origin, and also not completely sintered parts observed only in the composites containing in-situ prepared SiC(w).

### INTRODUCTION

According to the result of previous paper<sup>1)</sup>, with regard to the SiC whiskers(w), in-situ prepared in  $\text{Si}_3\text{N}_4$  powder(p) matrix, only the density was improved, and following two main problems remained unsolved; first of all, the diameter of SiC(w) thus obtained were found to be thinner than that of the industrial product perhaps because of the co-existing matrix interfering the whisker growth, and secondly, the strength of their composite was not superior to that of physically mixed composite.

It was then found that the whiskerizing reaction from silica and carbon black under pressure resulted in thick SiC(w)<sup>2)</sup>, which were, however, not obtained in-situ among the matrix.

In this article, efforts were made to solve these two items occurred in the in-situ procedure, and the fracture toughness was also determined, in addition to the strength measurement.

## EXPERIMENTAL PROCEDURE

## A. Materials

All of the starting materials,  $\text{SiO}_2$ , carbon black and  $\alpha\text{-Si}_3\text{N}_4$  (p) were the same as described elsewhere<sup>1)</sup>, but the sintering aids were changed to  $\alpha\text{-Al}_2\text{O}_3$  (TAIMICRON TM-D, Taimel Industries Company Ltd.) and  $\text{Y}_2\text{O}_3$  (Mitsubishi Chemical Industries Company Ltd.) from  $\text{La}_2\text{O}_3$  (Nissan Rare Earth Elements Chemical Company Ltd.) and  $\text{Y}_2\text{O}_3$  (P).

The catalyst to obtain thick SiC(w), having diameters of 1 - 2  $\mu\text{m}$ , was found to be a sort of iron powder, PR-350 (Kawasaki Steel Company Ltd.), the properties of which are summarized in TABLE I.

TABLE I. Properties of iron powder employed.

Chemical component	Total iron*	98.30 %
	Metallic iron*	94.67
Particle-size distribution (mesh)	> 100	trace
	> 145	trace
	> 200	0.1 %
	> 250	0.1
	> 350	1.2
	< 350	98.6
Density ( $\text{g/cm}^3$ )	2.29	

\* The difference between the both means the content of iron oxides.

B. Preparation of thickened SiC(w) in  $\text{Si}_3\text{N}_4$ (p)

Although it was found out that thickened SiC(w) were prepared under pressure<sup>2)</sup>, such as 0.35 MPa, they were scarcely observed among  $\text{Si}_3\text{N}_4$ (p) when the in-situ whiskerizing reaction was carried out without NaCl. Details of the in-situ whiskerizing procedure are the same as described elsewhere<sup>1)</sup>, except the reaction furnace. This furnace can be kept under pressure of lower than 0.5 MPa at the temperature of whisker formation, having a graphite heater in place of  $\text{LaCrO}_3$ , and also having a larger size being capable to produce 7 specimens for determination of physical properties. Without NaCl, the whiskers were formed mainly in the upper part on the matrix in the graphite crucible, although in-situ preparation of SiC(w) among the  $\text{SiO}_2$  matrix<sup>2)</sup> was observed, where the size of the  $\text{SiO}_2$ -matrix was as large as

several 10  $\mu\text{m}$ , being several 10 times larger than the  $\text{Si}_3\text{N}_4$  particles, having enough room in between to admit the whisker growth. However, in the presence of NaCl in the case of  $\text{Si}_3\text{N}_4$  matrix,  $\text{SiC(w)}$  were formed amongst the matrix submicron powder. The increase in the pressure during the reaction owing to the vaporization of NaCl, was not so remarkable, having made the reaction steadily safe, though it was necessary to control the pressure under 0.35MPa by occasional openings of the outlet of reaction system. To provide around 30 %  $\text{SiC(w)}$  in the mixture with the matrix,  $1700^\circ\text{C} \times 1.5\text{ h}$  was needed as a reaction condition, comparing with  $1600^\circ\text{C} \times 1\text{ h}$  in the case under normal pressure.

The reaction product thus obtained, was spread over a stainless screen, having the opening of 0.074 mm, under an air-flow of 7.0 l/min, at  $700^\circ\text{C} \times 5\text{ h}$ ,  $800^\circ\text{C} \times 3$  and  $5\text{ h}$ , being  $100 - 200^\circ\text{C}$  higher than the previous experiment<sup>1)</sup>, so that the trace amount of residual carbon can be eliminated more completely.

On the other hand, not a few trials to obtain thick  $\text{SiC(w)}$  under normal pressure were made, so that the reaction can be carried out by a continuous process, since the whiskerizing procedure under pressure was considered to be not so easy to realize it in a continuous system in practice. If the mechanism of  $\text{SiC(w)}$  formation could be elucidated as "VLS" (Vapor-Liquid-Solid) one, the root or core of every whisker-top must have a definite larger size to yield thick whiskers. The shortened ready-made thick  $\text{SiC(w)}$  having diameters of as large as 1-2  $\mu\text{m}$ , were employed as the seeds or cores of thick  $\text{SiC(w)}$ , but no effect to this purpose was observed in several runs of experiment. From these points of view, the iron having properties as shown in TABLE I, was found to be effective<sup>3)</sup>.

When the iron catalyst was used, the reacted product was immersed in 3 % HCl aqueous solution for 24 h to eliminate it.

#### C. Homogenizing by deagglomeration

After the air oxidation treatment, sintering aids were added in the ratio of  $\text{Si}_3\text{N}_4 : \text{Al}_2\text{O}_3 : \text{Y}_2\text{O}_3 = 89.5 : 3.00 : 7.50$  (wt %), followed by mixing with a tumbling mixer.

The tumbling mixer as described in previous paper<sup>1)</sup> was used, since this type of mixer was considered to be mostly appropriate for the purpose of preventing the whisker damage. About 300 ml of ethyl alcohol was added to about 50 g of the reaction product powder, which was mixed with plastic balls having a diameter of 15 mm, nylon-coated lead made, for 24 h in the mixer, followed by drying with a rotary evaporator under vacuum. For comparison, this mixer was used also in the case of physical mixing procedure.

#### D. Analysis of $\text{SiC(w)}$

After repeated investigations of X-ray diffractometry on a variety of mixtures of  $\text{SiC(w)}$  and  $\text{Si}_3\text{N}_4(\text{p})$ , the content of  $\text{SiC(w)}$  became able to determine quantitatively to a certain extent, as illustrated in FIG.1, having an excellent reproducibility, where the line was obtained by the minimum square procedure.

The condition of diffractometry was the same as described elsewhere<sup>1)</sup>.

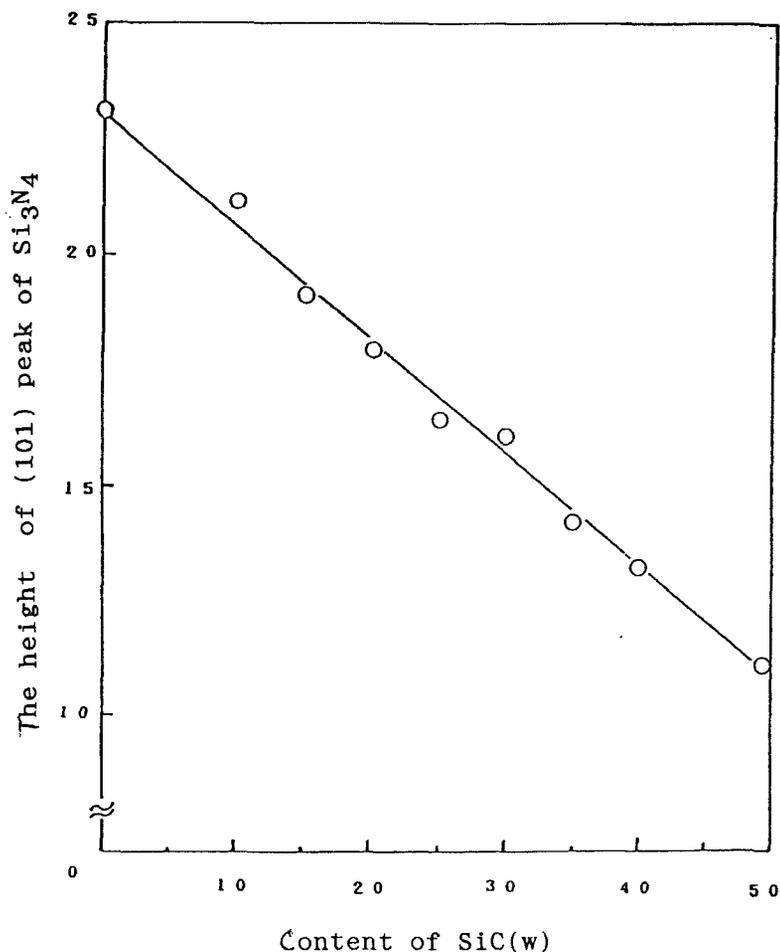


FIG.1. A relationship between the height of (101) peak of Si<sub>3</sub>N<sub>4</sub> and the content of SiC(w)

The content obtained by a gas analysis according to JIS R6124-1980, carried out by Kawasaki Refractories Company Ltd., was compared with that by diffractometry, as summarized in TABLE II, where the content of SiC(w) by gas analysis was calculated one by reducing the content of co-existing sintering aids, Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> analyzed by ICP method. The analyzed value of these sintering aids were approximately the same as the used amount, concerning 4 specimens in TABLE II.

TABLE II A Comparison of SiC(w) content between gas-analysis and X-ray diffractometry.

Mixing method	Physical				Chemical (in-situ)				
	No. of specimen	JB-5T*	JC-13T*	JC-15T*	JC-16T*				
Gas- analysis		35.8	34.2	61.0	38.0				
X-ray diffracto- metry		35.2	36.5	59.0	38.5				

\* See the foot note of TABLE III.

#### E. Fabrication of composite

Except the amount and sort of sintering aids, as above mentioned, every procedure to fabricate the composite by hot pressing, followed by machining, was the same as described in detail elsewhere<sup>1)</sup>. Relating to the physically mixed composites for comparison, the same tumbling mixer was also used.

#### F. Determination of density, strength and toughness

Differing from the previous work<sup>1)</sup>, the density of composite was determined by Archimedes method by employing distilled water. Concerning the bending strength, the same determination as above, in accordance with Japan Industrial Standard (J I S) by four point bending method at room temperature<sup>1)</sup>, was adopted.

Regarding with the determination of fracture toughness, S E N B (Single Edge Notched Beam)-method<sup>4)</sup> was used. The size of specimen was the same as that for measurement of the bending strength. At the center of specimen, a notch having the depth of 1.4 mm, was cut by a diamond cutter whose thickness was 0.2 mm. An Instron type (Autograph DSS-25T) was used for the determination, where the upper and lower spans were 13.3 and 40.0 mm respectively, and the cross head speed was 0.05 mm/min.

## RESULTS AND DISCUSSIONS

#### A. Thickening of in-situ prepared SiC(w) in Si<sub>3</sub>N<sub>4</sub>

As described in the section of experimental procedure, in-situ preparation of thickened SiC(w) in Si<sub>3</sub>N<sub>4</sub> under pressure, needed NaCl. This fact may suggest that the function of co-existing NaCl during the whisker formation was to provide the space enough to admit the whisker growth under pressure, owing to

the vaporization of NaCl having boiling point of  $1408^{\circ}\text{C}$ . According to several preliminary experiments to obtain thick SiC(w), the condition required for preparation of about 30 % (wt) of SiC(w) in the in-situ whiskerizing reaction product, was found to be severer, such as  $1700^{\circ}\text{C}$  x 1.5 h, comparing with  $1600^{\circ}$  x 1 h under normal pressure to prepare thinner whiskers. The pressure applied to the reaction, was prepared by CO gas, formed simultaneously with the whisker formation.

A typical example of in-situ prepared thick SiC(w) in  $\text{Si}_3\text{N}_4$  is as shown in FIG.2-(a), comparing with that under normal pressure in FIG.-2(b).

Because of the need to make the reaction condition severer, the thickening process under pressure must have been carried out by a slower crystallization, being enabled by the retardation of reaction, based upon Le Chatelier's principle. However, the process under pressure is obviously not so easy to make it continuous one in practice.

On the other hand, another successful result with the aid of iron catalyst under normal pressure, can be significant in an industrial sense. Now that the continuous process to produce the whiskers themselves is being carried on, this process can be applied to it with ease. Differing from the procedure to obtain thinner whiskers by employing 10 % aqueous solution of cobalt chloride<sup>1)</sup>, the iron powder having much larger size might form a larger liquid (L) mass as a root at the earliest stage in the V L S mechanism. The fact that the shortened thick SiC(w) could not behave as a core of crystallization to form thick SiC(w), having been found during this research work, might be an indirect proof of the V L S mechanism.

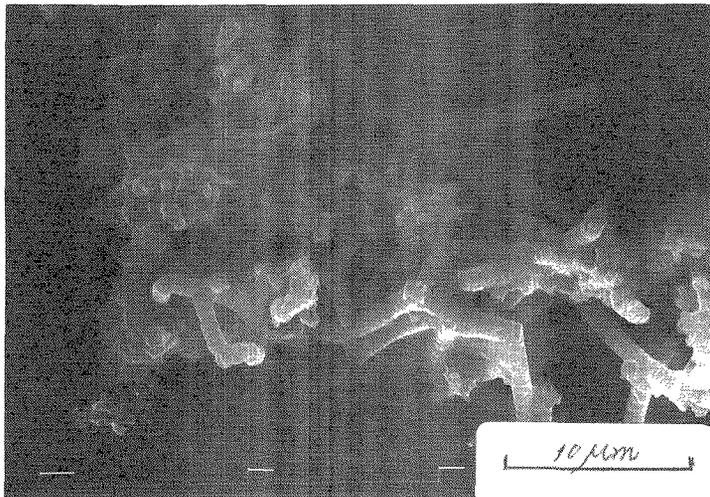


FIG.2-(a); Scanning electron microscopic (SEM) observation of SiC(w) in-situ prepared in  $\text{Si}_3\text{N}_4$  under 0.35 MPa

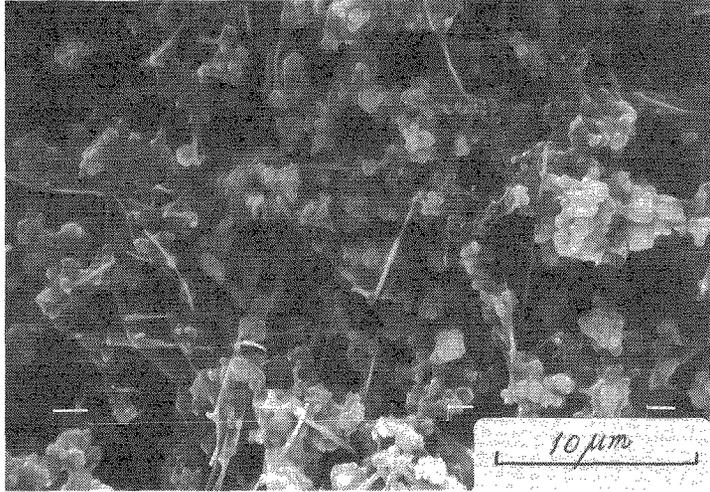


FIG.2-(b); SEM observation of the whiskers under normal pressure

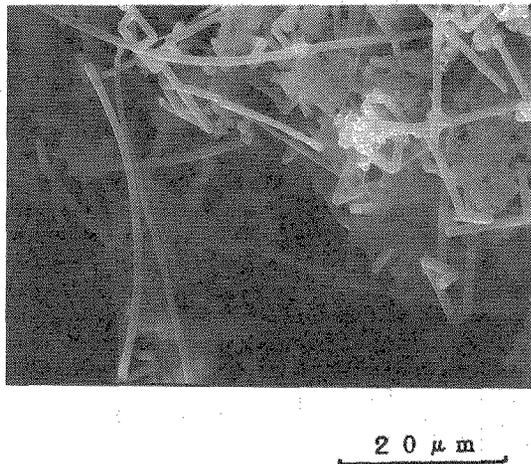


FIG.3; SEM observation of typical thick SiC(w), in-situ prepared in  $\text{Si}_3\text{N}_4$  with the iron catalyst under normal pressure

A typical photograph of in-situ prepared SiC(w) in the matrix is as shown in FIG.3.

However, the effect of the thickened SiC(w) thus obtained, to fracture toughness, was not recognized, in comparison with the thin ones as described later, so far as the toughness,  $K_{IC}$ , determined at room temperature is concerned.

## B. The physical properties of composites

TABLE III summarizes the density, bending strength and fracture toughness of representative composites in relation with the composition of starting materials.

## C. Discussions on the strength and toughness of composites

### (1) Bending strength

In comparison with one of the physically mixed composites, providing the highest bending strength (No.10 in TABLE III), every value of bending strength was found to be lower as seen in TABLE III. The residual carbon coarse particle of several  $10 \mu\text{m}$  in diameter, often observed as the fracture origin, was considered to play a big role to provide lower strength<sup>1)</sup>. However, it was found later that one of the fracture origins of No. 10 specimen providing the highest strength, 835 MPa, was also a carbon lump, confirmed by EPMA (Electron Probe Microanalyzer). This fact may suggest that there must be any other reasons why this type of composite, chemically mixed one, showed lower strength. In addition, the carbon particles observed in the specimens derived from the air oxidation at  $600^\circ\text{C}$  after the in-situ preparation of SiC(w), were not seen in those, oxidized at  $800^\circ\text{C}$ , perhaps because of more complete elimination of residual carbon.

To find out any other reason, many parts of composites, one chemically mixed specimen containing in-situ prepared SiC(w), No.7 in TABLE III, and the other one of physically mixed specimens, No.10 mentioned above, were observed in detail by optical microscopy. 5 parts in everyone of 6 - 7 specimens, prepared for the strength and toughness determination, were observed.

FIG.4 and 5 are the representative examples of the both, where FIG.4, chemically mixed one, was not the same as the other parts, in sharp contrast with FIG.5 showing almost the same homogeneous texture in all of the specimens observed. It should be also noted that the other physically mixed specimens, as No.11 and 12 in TABLE III, provided not so high properties as No.10. At any rate, one of the reasons providing lower strength might be poorer homogeneity of the chemically mixed composite. Several approaches to improve the defective homogeneity are being carried out.

TABLE III Physical properties determined at room temperature on the representative composites, together with the composition of starting materials.

Purpose	Specimen No.	Experimental No.	Raw materials (weight %)				
			$\text{Si}_3\text{N}_4(\text{p})$	C	NaCl	SiO <sub>2</sub>	Catalyst <sup>d)</sup>
Effect of thickness and length of SiC(w)	1	JC-22B <sup>a)</sup> (CK-S <sup>b)</sup> )	55.6	9.10	27.2	8.15	0
	2	JC-39T <sup>a)</sup> (CK-L <sup>b)</sup> )	55.6	9.10	27.2	8.15	0
	3	JC-25B (N-S <sup>b)</sup> )	55.5	9.10	27.2	8.14	0.10
	4	JC-24T (N-L <sup>b)</sup> )	55.5	9.10	27.2	8.14	0.10
	5	JC-52T (CK-L <sup>b)</sup> )	55.5	9.10	27.2	8.14	0.10*
	6	JC-53B (CK-S <sup>b)</sup> )	55.5	9.10	27.2	8.14	0.10*
	7	JC-56T (N-L <sup>b)</sup> )	55.5	9.10	27.2	8.14	0.10
	8	JC-57B (N-S <sup>b)</sup> )	55.5	9.10	27.2	8.14	0.10
Comparison	9	JA-6T <sup>a)</sup> (monolithic)	—	—	—	—	—
	10	JB-8T <sup>a)</sup> (physically mixed)	—	—	—	—	—
	11	JB-6S <sup>c)</sup> (physically mixed)	—	—	—	—	—
	12	JB-51B (physically mixed)	—	—	—	—	—
Control of SiC(w)-content	13	JC-37T	59.7	5.33	25.9	4.86	0
	14	JC-40T	64.1	7.32	22.0	6.61	0
	15	JC-35T	55.6	9.10	27.2	8.15	0
Elimination of trace C	16	JC-42T	55.5	9.10	27.2	8.14	0.10*
	17	JC-44T	55.5	9.10	27.2	8.14	0.10*
	18	JC-48T	55.5	9.10	27.2	8.14	0.10*

a) J; JIS-size, A; monolithic (non composite), B; physically mixed, C; chemically mixed, -T; by tumbling mixer (longer whiskers), -B; by ball mill (shorter whiskers)

b) CK; thick SiC(w), N; thin SiC(w), -L; longer, -S; shorter

c) S; homogenized by suspension<sup>1)</sup> d)  $\text{CoCl}_2$  aq. except \* iron powder, 0 means under pressure of 0.35 MPa without catalyst

TABLE III(cont'd)

SiC(w) %	n	Relative density,%		Flexural strength,MPa			Fracture toughness,MPam <sup>0.5</sup>			Condition of air- oxidation
		X	s	n	X	s	n	X	s	
52	7	96.0	0.93	4	439	79	3	4.84	0.02	600 x 15
35	7	98.8	0.21	4	437	140	3	5.79	0.37	600 x 15
33	7	98.9	0.04	4	447	22	3	6.47	0.42	600 x 15
30	8	100	0.08	4	445	29	3	7.81	0.29	600 x 15
27	7	95.7	0.06	4	439	23	3	6.05	0.18	800 x 3
27	6	98.1	0.09	3	652	5	3	5.86	0.13	800 x 3
32	7	98.9	0.47	4	446	37	3	6.01	0.09	800 x 3
33	6	97.9	0.21	3	427	46	3	6.01	0.06	800 x 3
0	7	99.4	0.08	4	412	43	3	6.99	0.20	—
30	4	97.6	0.006	4	835	76	3	9.28	0.55	—
32	7	95.5	0.008	4	455	25	1	7.01	—	—
30	6	99.4	0.14	3	766	19	3	6.90	0.06	—
12	7	99.0	0.14	4	437	140	3	5.79	0.37	600 x 15
19	7	99.2	0.14	4	692	88	3	7.11	0.63	600 x 15
32	7	99.3	0.20	4	271	30	3	5.34	0.93	600 x 15
23	7	98.9	0.09	4	466	33	3	5.98	0.08	700 x 5
27	6	98.4	0.27	3	587	11	3	6.85	0.05	800 x 3
27	5	98.4	0.17	3	434	40	2	5.98	0.16	800 x 5

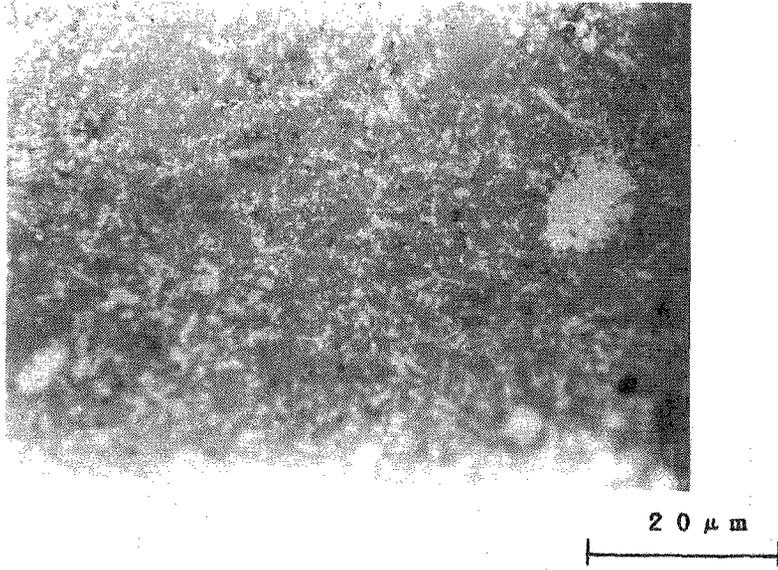


FIG.4; A typical texture of chemically mixed composite by optical microscopy

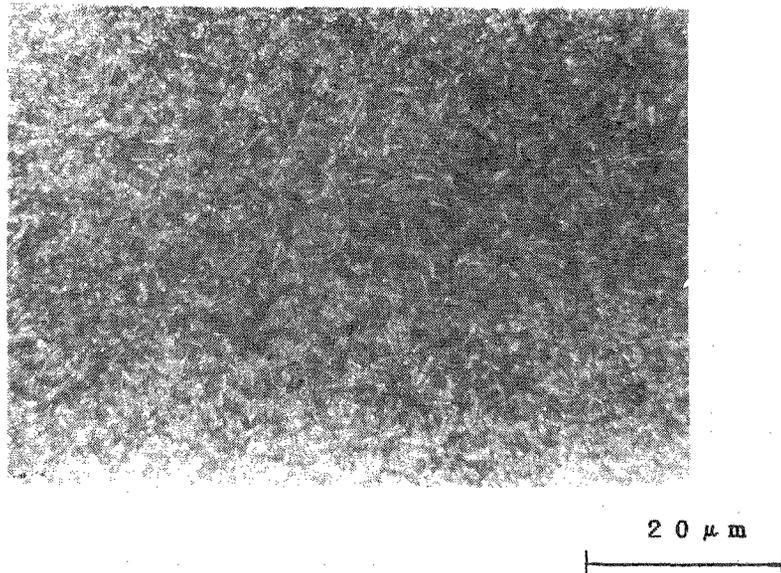


FIG.5; Homogeneity in the texture of physically mixed composite providing the highest strength and toughness

## (2) Fracture toughness

Concerning the fracture toughness determined by S E N B method, No.10 specimen, one of the physically mixed composites, having the highest strength, provided also the highest toughness. It is not sure that the homogeneity as above mentioned with regard to this specimen is a decisive element to provide the high physical properties, but the aim of this research by preparing SiC(w) in-situ in matrix lay in improving the homogeneous dispersion of the whiskers, so that the physical properties can be raised. Any lumpish material appeared at the fracture origin being peculiar to the in-situ prepared composite, must not exist, since it may play a big role to lower the strength and toughness. In the case of physical mixing, both of the whiskers and matrix powder are refined materials in industry.

The part where the matrix did not completely sintered, were observed only in the chemically mixed composite, as shown in FIG.6. Such a part must have brought about lower physical properties, although the reason why it was seen only in the composites containing in-situ prepared SiC(w), was not clear. It can be considered that the sintering aids were relatively more difficult to come near the matrix due to more closely formed SiC(w) to the matrix powder, comparing with the physically mixed composite.

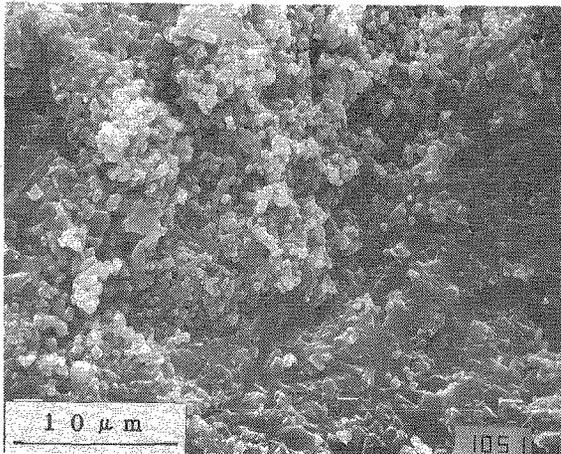


FIG.6; Not completely sintered part in the composite of No.8 in TABLE III  
Standing at the general view-point, a relationship between

the bending strength and fracture toughness can be summarized in general, as illustrated in FIG.7, which is one example within a range of published values in literatures<sup>5)-12)</sup>, although the determining method, SiC(w) content and the other condition are different from each other. One can estimate the position of the properties reported in this article. Even if the properties of chemically mixed composite cannot be improved comparing with those of physically mixed one, the former process may be of significance in the sense of improving the working environment at the whisker producing factory.

On the other hand, it should be also noted that a mullite composite including in-situ prepared whiskers of mullite showed much higher fracture toughness<sup>13)</sup> than that of physically mixed composite, though the strength was not determined. In this case, the mullite whiskers can be formed only by the most appropriate heat-treatment, not by any chemical reaction from several sorts of starting materials, where there may be much less possibility to form coarse lumpish materials, as well as to form heterogeneous texture of the relevant composite.

Consequently, without making efforts to improve the homogeneity as well as to remove the coarse particle in this in-situ process, no improvement of physical properties can be expected. In-situ whisker growth during the sintering process would be desired in this sense, as an example of self-reinforced or self-formed composite, where the accurate control of SiC(w)-content as well as their size should be possible.

### (3) The other discussions

Concerning the condition of air oxidation to eliminate the residual trace carbon, the physical properties of composite was not affected by it, as seen in TABLE III. As above mentioned, no carbon particle was observed at the fracture origins in many specimens treated by air oxidation at 800°C prior to hot pressing. Accordingly, the residual carbon particle might have not caused the lower properties.

With regard to the control of SiC(w)-content as another problem of this in-situ procedure, it was found to be possible as observed in No.13 - 15 in TABLE III, by changing the composition of starting materials.

There remains another question; whether  $\text{Si}_3\text{N}_4$ -matrix may change chemically or not during the whiskerizing reaction at 1600°C for 1 h. From this view-point, only the matrix powder was heat-treated under the same whiskerizing condition as above. No change in X-ray diffractometry before and after this heat-treatment was observed, whereas a small amount of whiskers was observed in S E M of the heat-treated powder. Density of the both hot pressed specimens were 97.3 and 98.8 %, before and after the treatment respectively; while the bending strength of the both were 725 and 842 MPa, and the fracture toughness were 7.77 and 7.74 MPam<sup>1/2</sup>, respectively. It could be thus made clear that no degradation of the matrix powder itself occurred during the heat-treatment, required for the in-situ preparation of SiC(w) in the matrix.

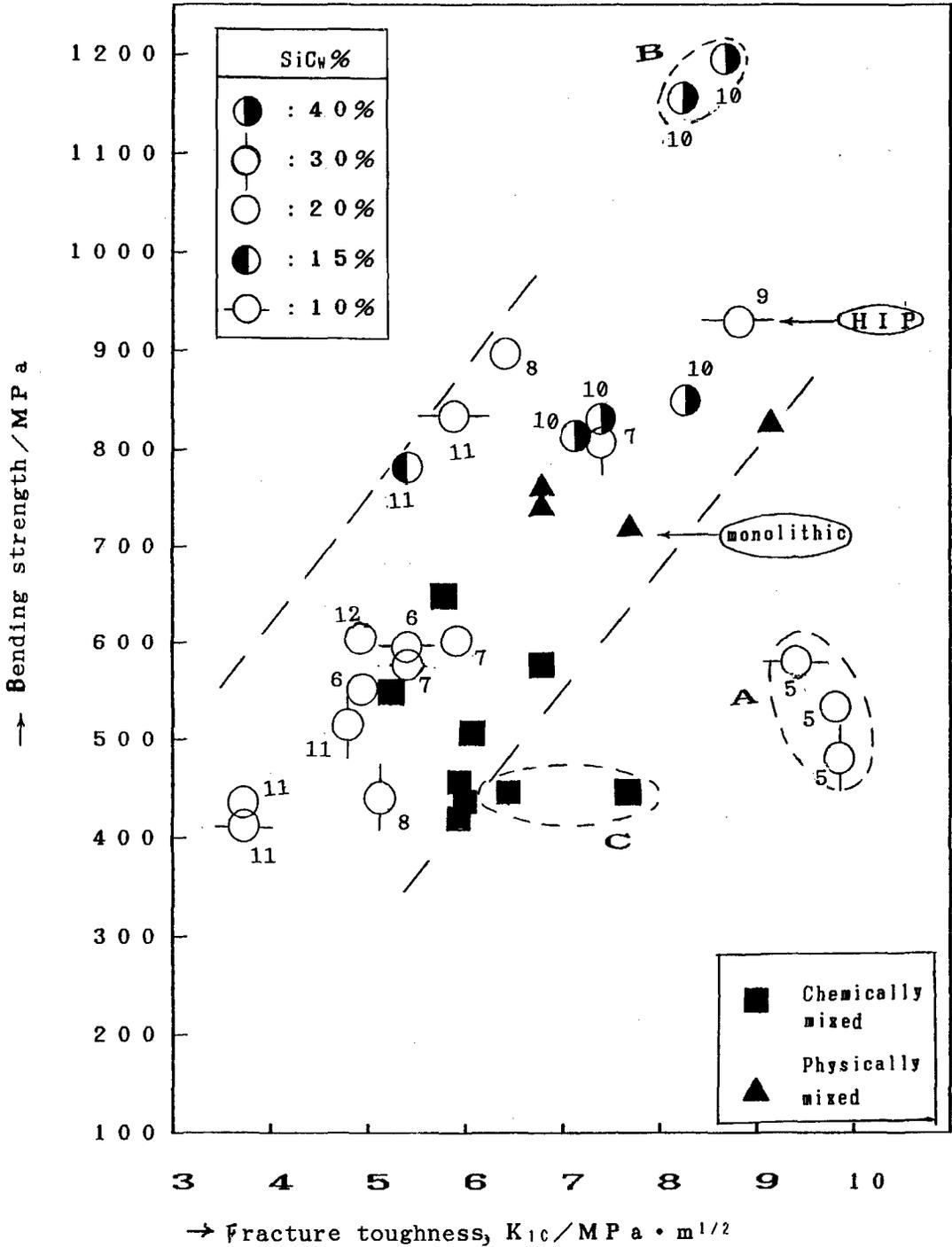


FIG.7; Relation between bending strength and fracture toughness, where A-group used thicker(3-10  $\mu\text{m}$ ) whiskers,B-group used a large amount of sintering aid, and C-group was irregularly machined. The number near the round mark corresponds to the reference No. cited in the last part.

## CONCLUSION

Thickening procedure of thin SiC(w), in-situ prepared in Si<sub>3</sub>N<sub>4</sub> matrix under pressure, needed co-existing NaCl to admit the whisker growth among the matrix powder. The same purpose under normal pressure was fulfilled by employing iron powder having a particular size.

Strength and toughness of the composite containing in-situ prepared SiC(w), were not so high as the most homogeneous composite that was physically mixed. Improvement of homogeneity and elimination of coarse particle as the fracture origin, were considered to be needed for strengthening and toughening of chemically mixed composite, containing in-situ prepared SiC(w). From the standpoint of environmental problem in the whisker treatment, the in-situ process must have a positive meaning.

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