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Carderock Division
Naval Surface Warfare Center
Bethesda, MD 20084–5000

CDNSWC–SME–92–10 March 1992
Ship Materials Engineering Department
Research and Development Report

Effect of Whisker Orientation on the Mechanical Properties of Silicon Carbide/Alumina Composites

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A. Srinivasa Rao

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TABLES

1. Alumina powder and silicon carbide (whisker) characteristics.

2. Flexural strength and fracture toughness of sintered SiC / Al_2O_3 composites with and without the whisker orientation.

ABSTRACT

Silicon carbide (whisker) and alumina composites were processed with and without concurrent application of an external electric field in order to orient the whiskers in the composites. Both the flexural strength and the fracture toughness of the composites were measured. The results suggest that the application of electric field improves the whisker orientation of the sintered composites. However, the improvement in the mechanical properties of the composites due to the orientation of the dispersed whiskers is not very significant (~ 9-12 %). Samples processed from flocculated suspensions produce poorly dispersed microstructure.

ADMINISTRATIVE INFORMATION

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INTRODUCTION

Ceramic matrix composites have been the focus of recent investigations because of their potential use in the fabrication of structural components used at high temperatures. Although in the open literature, most of the work has been reported on the processing of either the fiber or whisker or particulate reinforced ceramic matrix composites [1-4], except for few reports [5-6], very little has been reported on the effect of the dispersed particle or whisker shape or orientation on the mechanical properties of the composites. The preparation of particulate or whisker reinforced ceramics requires the uniform distribution of the filler material throughout the matrix, in order to avoid the agglomeration and hence the oversized particles.

It has been well established that different ceramic powders can be homogenized via a dispersion phase and with proper control of zeta potential [7] or by the coprecipitation of second phase onto the surface of the matrix phase [8]. Since non - spherical particles such as the whiskers tend to form mechanical bridges during the mixing process [9], and also cause a serious problem during the filtration process, a simple approach of slip derived ceramic processing may be inadequate in maximizing the mechanical properties of sintered ceramic composites. The present investigation was undertaken in order to study the effect of whisker orientation on the mechanical properties of the silicon carbide whisker reinforced alumina ceramics.

MATERIALS

Commercially available alumina powder (A-16 SG from Alcoa Aluminum Company Co., Pittsburgh, Pa) and silicon carbide whiskers (Tateho Chemical Co., Japan) were used in the preparation of the composite materials. The physical characteristics of the powder and whiskers supplied by the manufacturers is given in Table 1.

EXPERIMENTAL PROCEDURE

a). Processing of silicon carbide (whiskers)/alumina composites

The silicon carbide/alumina composites were processed as follows : Firstly, alumina and silicon carbide slips were prepared by dispersing predetermined amounts of silicon carbide whiskers (0-20 vol.%), and alumina powder in water using an ultrasonic probe for 10 min. Once these dispersions were prepared, the mixture was sprayed under high pressure in order to ensure a thorough mixing of the whiskers and powder in suspension. It has to be pointed out that due care was taken to prevent any breaking of brittle whiskers during the high pressure spray mixing operation. The suspension was then introduced into a special filtration cell and was subjected to a filtration process. The filtration cell is a simple filter press that has been modified in order to influence the direction of transport of a given particle or whisker during the settling process. Although, the actual details of the filtration cell are proprietary, a schematic diagram of the cell is shown in Figure 1. The filtration cell is a low pressure filter press that has been provided with two electrodes at both ends of the cell. An applied dc potential across the two electrodes provides the required electric field to the settling particles during the filtration process.

The filtered green ceramic composite specimen containing directionally oriented whiskers and alumina particles were dried in an oven maintained at 80°C for 24 h. The dried green samples were sintered in a furnace at 1600°C for 10 h in an inert atmosphere and then cooled. In order to minimize the thermal stresses due to the release of water during heating, the green samples were first heated at the rate of 2°C min^{-1}, until the furnace had reached 200°C. After initial baking is over, the furnace temperature was raised to 500°C at the rate of 2°C min^{-1}. The samples were heat treated for 2 hours at 500°C; later these samples were heated at the rate of 3°C until the furnace had reached the sintering temperature (1600°C).

All sintered samples were polished and a number of small bars were cut for the determination of the density, flexural strength, toughness and the microstructure.

b). Electrophoresis measurements

In order to determine the optimum pH conditions for obtaining stable and low viscosity silicon carbide (whisker)/alumina suspension, the electrophoresis experiments were carried out. Dilute suspensions (~ 1 wt. %) of silicon carbide (whisker), alumina and silicon carbide/alumina mixtures were prepared by dispersing the ceramic powders/whiskers in 10^{-3} mol dm^{-3} KCl solution using a magnetic stirrer. The pH of the dispersions was adjusted by adding small amounts of 1 mol dm^{-3} HCl or NaOH solution followed by thorough mixing of the suspension. The zeta potential of the dispersed particles was determined using microelectrophoresis apparatus.

c). Measurements

The density of the sintered composite samples was determined using the Archimedes principle. The flexural strength of the composites was measured using the four point bend test and the microstructural details were analyzed using scanning electron microscopy.

RESULTS

The zeta potential versus pH profiles of silicon carbide (whisker), alumina and their mixtures in dilute dispersions are shown in Figure 2. These results indicate that both silicon carbide and alumina surfaces are negatively charged above pH 9. However, the situation with the mixture tends to depend upon the concentration of each constituent. This information is very important because we do not want to create a situation for the alumina and silicon carbide particles to migrate in different directions during filtration process. It was realized that although the information obtained from zeta potential estimates provides useful guidance in an ideal situation such as the filtration of pure silicon carbide or alumina, interpretations based on only the zeta potential results are inadequate in determining the filtration behavior of the mixture. This is because, the hetero-flocculation of the system is not considered in the zeta potential measurement.

In order to estimate the floc (agglomerated particle) size of the dispersed particles in suspension, a few sedimentation experiments were carried out. The mean floc diameter of the particles was estimated from the sedimentation data using the modified Steinour equation [10]. The results obtained on the normalized floc diameter (diameter of the floc estimated from sedimentation experiment/actual mean diameter of the particle or the whisker) are shown in Figure 3. Since it is very difficult to measure the diameter of the whiskers, it was assumed in the present calculations that the ultimate mean diameter of the particle or the whisker is equal to the value that has been estimated from the measurement of the total surface area. The results shown in Figure 3 suggest that hetero-flocculation of the silicon carbide and alumina particles in suspension occur in the pH range pH 3.5 - 9.0 and above pH 9.0 or below pH 3.5 the suspensions of silicon carbide and alumina are very stable.

Figure 4 shows the sintered composite density versus silicon carbide concentration plots obtained for three different sets of samples. The results suggest that the densification of the composites depend upon the whisker concentration of the composite and the state of the suspension (pH). The results also indicate that the application of electric field has improved the final composite density. However, the magnitude of the increase in the sintered composite density due a change in the pH of the slip and the whisker orientation appears to be small (1 - 2 %).

The flexural strength and fracture toughness of the composites is shown in Table 2. From Table 2 it can be observed that the composites produced from stable suspensions are stronger than the composites produced from flocculated slips. For example, the composites produced from slips at pH 9.5 (stable suspension of silicon carbide and alumina : Figures 2 and 3) have nearly 5 % greater strength and fracture toughness than the composites produced from slip at pH 5 (flocculation of silicon carbide and alumina particles due to hetero-flocculation : Figure 3). From the results shown in Table 2 it can also be noticed that the flexural strength of the composites containing oriented whiskers is ~ 9 - 12 % higher than the total strength of the composites that were produced using the conventional processing method. Similar trends are also discernible for the fracture toughness of the composites.

From a number of electron micrographs obtained from all sintered composite samples, we found that the orientation of the whiskers has improved considerably, because of the application of an electric field across the filtration cell. The microstructure of the samples produced from flocculated suspensions often showed poor dispersibility of the whiskers in the matrix due to the presence of large agglomerates. A typical microstructure of the fractured surface of sintered composites containing 15 vol.% silicon carbide is shown in Figure 5. Figures 5(A) and (B) represent the microstructure of the fractured surface of whisker oriented composites processed from stable (pH 9.5) and flocculated (pH 5) suspensions respectively and Figure 5(C) represents the fractured surface of an un oriented sample processed at pH 9.5. The direction of fracture in all these whisker oriented composites is at right angles to the whisker orientation.

DISCUSSION

The above results clearly indicate that fine whiskers can easily be oriented in a specific orientation due to the application of an electric field during the filtration process. However, the results suggest that the microstructure and properties are dependent on the state of the slip. During sintering, the loosely held matrix particles in green composites tend to coalesce and grow to form large grains. If the particles and the whiskers are flocculated, the sintering process may produce either large grains or porous materials. Such a sintering degrades the mechanical strength of the composites.

Although, one can explain the degradation of the mechanical properties of composites processed from flocculated suspensions, in terms of the inhomogeniety of the sintered composite microstructure, it is not clear why an improvement in the orientation of whiskers did not significantly increase the mechanical strength and toughness of the composites. In general the sequence of events for the failure of ceramic matrix composites that has been suggested in the literature can be summarized as follows : When a ceramic matrix composite is subjected to mechanical deformation, after a critical stress level is reached, fine cracks are initiated along the edges of the composite. These cracks can progress across the composite surface along the interface between the matrix and the filler, or can traverse right through the filler particles (if the strength of filler material is much lower than the matrix), till the composite fails. It was also suggested that if the cracks in a composite are deflected during the crack propagation process, (either by blunting the cracks or by increasing the path length), then the material will fail at a higher stress (load) level.

If one applies a similar mechanism for the two sets of composites processed here (viz. SiC whisker oriented composites versus SiC whiskers randomly dispersed composites processed at pH 9.5), one would predict a longer crack length for the failure of the later case than the former case. A schematic diagram of the sequence of such crack initiation and propagation in whisker oriented composites is given in Figure 6. Figure 6 (A) shows a cross section of the whisker oriented and randomly dispersed composites (with the whisker orientation perpendicular to the direction of the applied load). Once the composites are stressed above a critical level fine cracks tend to nucleate along the edges of the composite (Figure 6(B)). A continued increase in the applied load, can accelerate the progression of the cracks along the interface between the matrix and the whisker. The total path length of the composites with oriented whiskers would be shorter than the composites with randomly dispersed whiskers (Figure 6(C)). Hence, the composites with whiskers randomly oriented will fail at a higher stress (load) level. Since our present results suggest that the flexural strength and fracture toughness of whisker oriented composite is slightly higher than the randomly oriented composites, it is possible that an additional parameter, such as the composite density, (the density of oriented composites is ~ 1-2 % higher than the randomly oriented composites) may also play some important role in the improvement of the mechanical property of whisker reinforced composites.

In order to derive positive conclusions, and attribute the differences in SiC whisker reinforced alumina composite flexural strength and toughness to the composite density and crack deflection ability of random orientation of the whiskers unequivocally, some additional experiments have been planned for the future.

CONCLUSION

From the present investigation, the following conclusions can be derived :

1. Both flexural strength and fracture toughness of alumina composites can be improved by adding silicon carbide whiskers in the concentration range 0 - 20 vol.%.
2. The concurrent application of an electric field during filtration process improves the orientation of whiskers in the sintered composites.
3. The increase in the composite mechanical properties due to whisker orientation depends upon the pH of the filtered suspension.
4. Physical alignment of whiskers of the composites alone is not sufficient to improve the mechanical properties

significantly. It appears that some random orientation of the whiskers is required for mechanical strength and toughness.

REFERENCES

1. K. M. Prewo and J. J. Brennan, "High Strength Silicon Carbide Fiber Reinforced Glass - Matrix Composites," J. Mater. Sci., 15, 463 (1980).

2. I. W. Donald and P. W. Mc Millan, "Review : Ceramic Matrix Composites," J. Mater. Sci., 11, 949 (1976).

3. F. D. Gac, J. V. Milewski, W. J. Parkinson and J. J. Petrovic, "Short Fiber Reinforced Structural Ceramics," pp. 325 in Fossil Energy Program Quarterly Progress Report, ONRL / FMP-83-2, Oak Ridge National Laboratory, Oak Ridge, TN (1983).

4. F. F. Lange, "Transformation Toughening," J. Mater. Sci., 17, pp 225, 235, 240, 247 (1982).

5. G. C. Wei and P. F. Becher, "Development of SiC - Whisker Reinforced Ceramics,"Bull. Amer. Ceram. Soc., 64[2], 298 (1985).

6. P. F. Becher and G. C. Wei, "Toughening Behavior in SiC - Whisker Reinforced Alumina," Comm. Amer. Ceram. Soc., 63[12], C-267 (1984).

7. S. Baik, A. Bleier and P. F. Becher, "Preparation of Al_2O_3 - ZrO_2 Composites by Adjustment of Surface Chemical Behavior," Better Ceramics Through Chemistry,

8. A. S. Rao and W. R. Cannon, "Alumina and Zirconia Composites Produced from Commercial Alumina and Synthesized Hydrous Zirconia," Ceramics International, 15, 179 (1989).

9. P. A. Hartley, "Cohesive Strength of Powder Compacts," Ph. D. Thesis, Carnegie - Mellon University, Pittsburgh, Pa (1986).

10. D. Dollimore and K. Karimian, "Sedimentation of Suspensions : Factors Affecting the Hindered Settling of Alumina in a Variety of Liquids," Surface Technology, 17, 239 (1982).

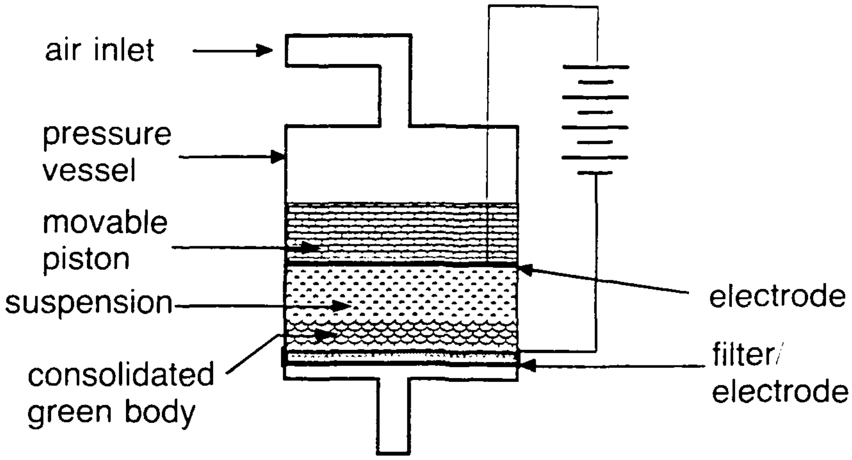

Figure 1. Schematic diagram of the electric field assisted filtration cell

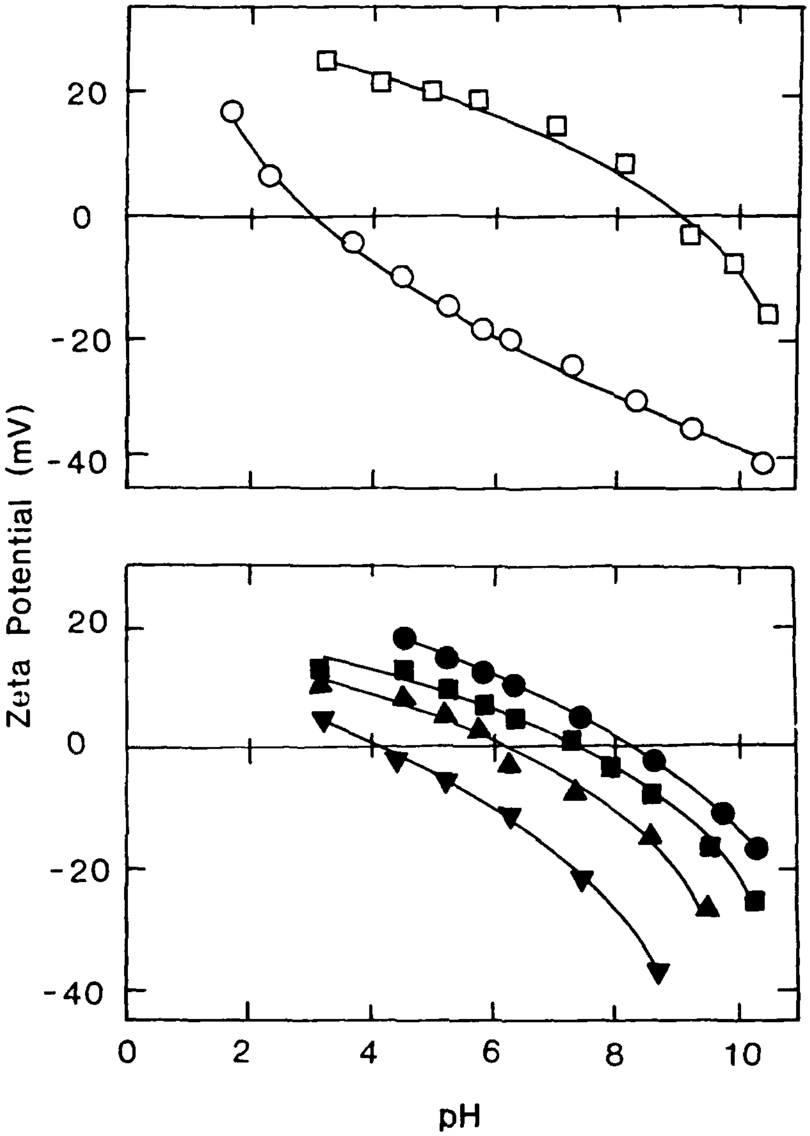

Figure 2. Zeta potential versus pH of (A) (○) silicon carbide and (□) alumina and (B) (● ■ ▲ ▼) silicon carbide / alumina mixture dispersed in 10^{-3} mol dm^{-3} KNO$_3$ solution. Silicon carbide concentration (●) 5, (■) 10, (▲) 15 and (▼) 20 vol.%.

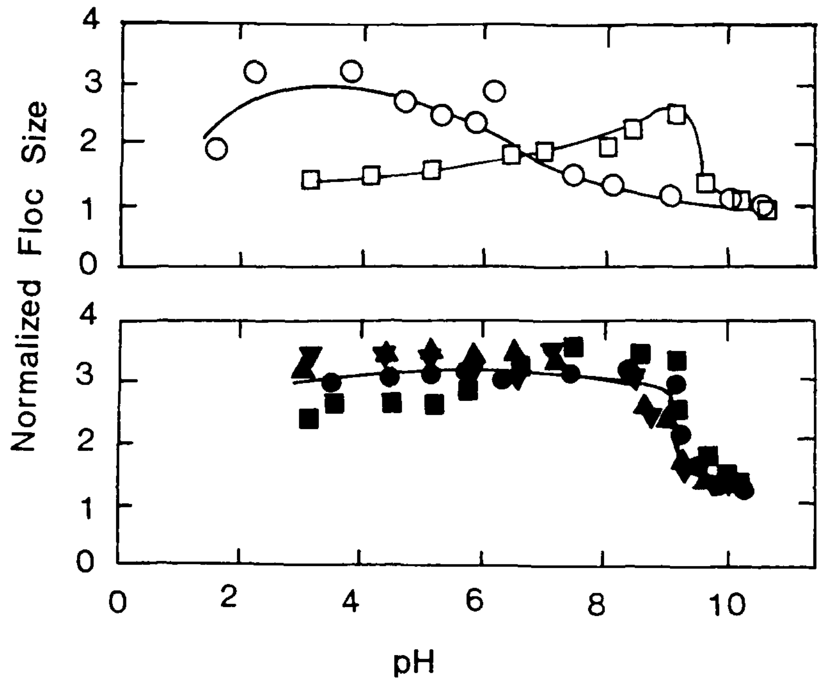

Figure 3. Normalized floc size versus pH of (A) (○) silicon carbide and (□) alumina and (B) (● ■ ▲ ▼) silicon carbide / alumina suspensions. Silicon carbide concentration (●) 5, (■) 10, (▲) 15 and (▼) 20 vol.%.

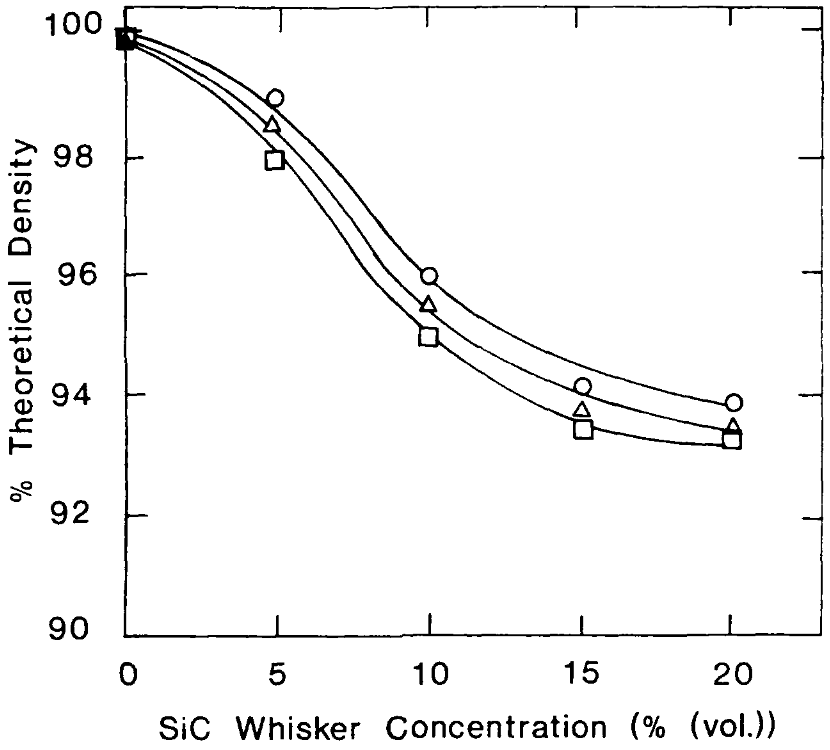

Figure 4. Sintered silicon carbide whisker reinforced alumina composite density versus silicon carbide concentration as a function of process methodology. Processing condition : (○ □) concurrent application of electric field and (△) no electric field during filtration of the slip. pH of the slip (○ △) 9.5 and (□) 5.0.

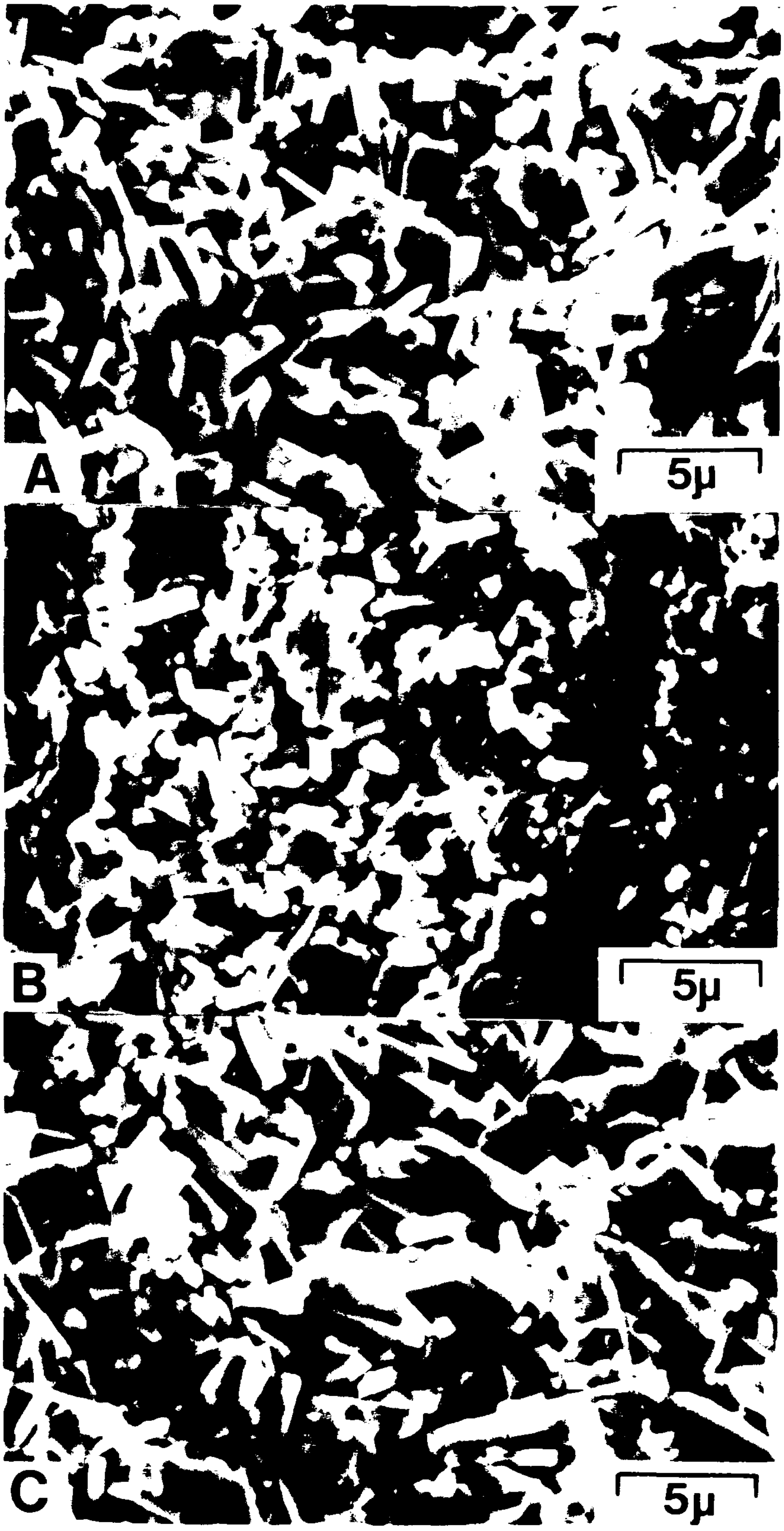

Figure 5. Scanning electron micrograph of the fractured surface of sintered silicon carbide / alumina composite as a function of process methodology. Processing condition : (A,B) concurrent application of electric field and (C) no electric field during filtration of the slip. pH of the slip (A,C) 9.5 and (B) 5.0. Silicon carbide concentration 15 vol.%.

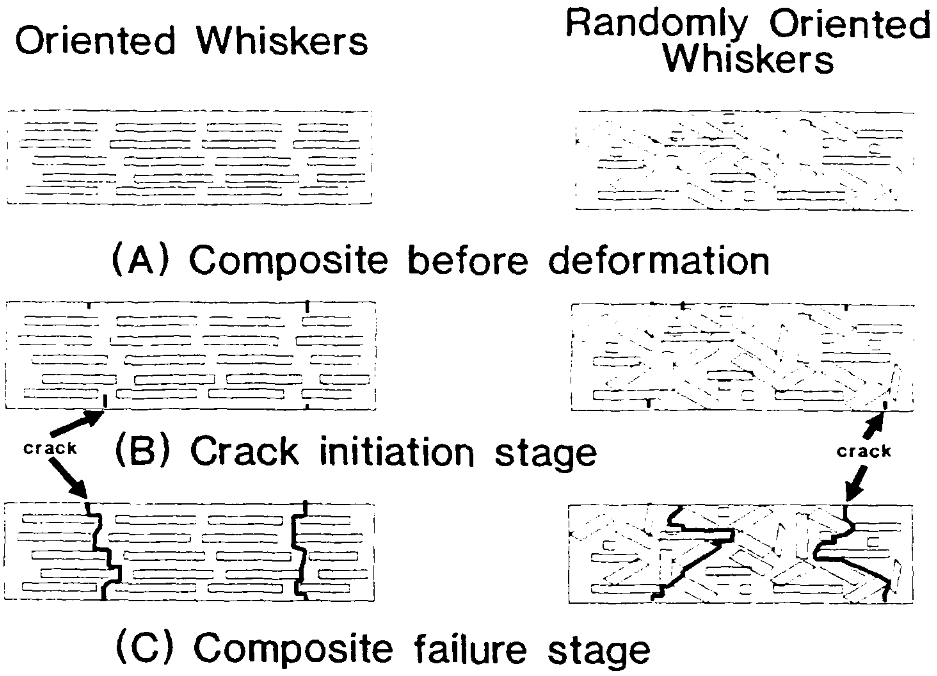

Figure 6. Schematic diagram of the crack initiation and progression in a whisker reinforced composite during deformation.

Table 1. Alumina Powder and Silicon Carbide (Whisker) Characteristics

Powder/Wisker Characteristics	A-16 SG Alumina	Silicon Carbide Whiskers
Mean Diameter (µm)	2.0	0.4
Density (gm/C C)	3.98	3.19
Surface Area (m^2/gm)	10.2	
Whisker Diameter (µm)		0.1 - 0.5
Whisker Length (µm)		30 - 100
Aspect Ratio		50 - 200
Whisker Strength		3 - 14 GPa
Crystal Phase	α - Al_2O_3	ß - SiC 95% α - SiC 5%

Table 2. Flexural Strength and Fracture Toughness of Sintered SiC/Al_2O_3 Composites With and Without the Whisker Orientation

Whisker Concentration	Flexural Strength (MPa)			Fracture Toughness (MPa $m^{1/2}$)		
	No. Orientation	Oriented Whiskers		No. Orientation	Oriented Whiskers	
	pH	pH		pH	pH	
	9.5	5.0	9.5	9.5	5.0	9.5
0	330	336	340	4.2	4.1	4.2
5	320	340	362	4.7	5.0	5.4
10	356	380	398	5.5	5.8	6.0
15	380	396	415	7.1	7.3	8.0
20	386	410	427	6.8	6.9	7.3

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Silicon carbide (whisker) and alumina composites were processed with and without concurrent application of an external electric field in order to orient the whiskers in the composites. Both the flexural strength and the fracture toughness of the composites were measured. The results suggest that the application of electric field improves the whisker orientation of the sintered composites. However, the improvement in the improvement in the mechanical properties of the composites due to the orientation of the dispersed whiskers is not very significant (9-12%). Samples processed from flocculated suspensions produce poorly dispersed microstructure.

ALUMINA, SILICON CARBIDE WHISKERS
PROCESSING STRENGTH TOUGHNESS

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