# A COST-EFFECTIVE FABRICATION METHOD FOR CONTINUOUS FIBER CMCS WITH EXCELLENT FRACTURE TOUGHNESS

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## Abstract

Si-Zr-C-O long fiber /$Al_2O_3$ laminated composites have been successfully developed using pre-impregnated (prepreg) sheets. The monolayer prepreg sheets were prepared via infiltration of alumina/glass aqueous slurry into aligned long fibers, then laminated and finally pressureless-sintered in the furnace (in air).
The key of this process is to add the $SiO_2$-$B_2O_3$-based glass powder into alumina slurry in order to avoid the degradation of fibers during sintering of the fiber contained green body.

The purpose of this work is to investigate the influence of alumina/glass weight ratio, sintering temperature and sintered density affecting the mechanical properties such as bending strength and fracture toughness. The Si-Zr-C-O long fiber /$Al_2O_3$ composite sintered at 900 ºC in alumina/glass weight ratio 6/4 showed the maximum bending strength of 405 MPa and fracture toughness of 20 MPa·m$^{1/2}$, which suggests prospective applications to various structures (aerospace, automotive and others) under high temperature environment.

## 1 Introduction

Ceramic matrix composites (CMCs) have been developed to overcome the inherent low fracture toughness of ceramic materials. It is well-known that long fiber reinforced ceramics is most effective method to fulfill high toughness of ceramics. Much of the past effort has been concentrated on understanding toughening, the role of interfacial adhesion, and the development of novel fabrication techniques [2]. However, the techniques developed so far often produce matrix poor regions, or possess a problem in fiber/matrix interfacial characteristics due to degradation of fibers during processing and thus these materials do not meet the needs of demanding applications in terms of the material uniformity, reproducibility, superiority to monolithic ceramics and cost effectiveness.

The author developed a low-cost production route to CMC [3]. The fabrication process does not require any expensive fabrication facilities and is concluded to be cost-effective and have a good potential for significantly increasing mechanical properties such as static strength, fracture toughness, and fatigue resistance. The key of this process is to add the $SiO_2$-$B_2O_3$-based glass powder into alumina slurry in order to avoid the degradation of fibers during sintering of the fiber contained green body.

## 2 Fabrication Method and Experimental Procedures

The fabrication process is shown in Fig.1. The aqueous homogeneous slurry was prepared by ball milling the mixture of $Al_2O_3$ powder (α-alumina AL45-1, Showa Denko Co. Ltd, Japan) and $SiO_2$-$B_2O_3$ based glass powder (SNK-01, Senyo Glass Co. Ltd, Japan) and additives (binder, plasticizer, dispersant, ethanol, distilled water, etc) in a polyethylene jar using yttria stabilized zirconia milling media. The average particle size of $Al_2O_3$ was 1.1μm and $SiO_2$-$B_2O_3$-based glass was 4.0μm. The copolymer of acrylic acid and acrylic acid ester (AQ-2559, Lion Corporation, Japan) was used as binder and dispersant agent. The reinforcing material chosen for this study was Si-Zr-C-O fibers (ZMI-S1E08PX, Ube Industries Ltd, Japan). In this work, the simplified casting method based on a tape casting technique was utilized to prepare pre-impregnated (prepreg) sheets. The aqueous slurry was vacuum defoamed then cast uniformly on uniaxial aligned fibers setting on a coater. Then the cast fibers were vacuum-assisted to infiltrate the slurry into the multifilament Si-Zr-C-O fibers. The thickness of prepreg sheet was determined by adjusting the clearance between coater and roller. The prepreg sheets were dried to remove the solvent. Monolayer pieces were then cut into specimen geometry, and laminated together to prepare the unidirectional multilayer-preforms.

The multilayer-preforms of fiber-containing prepreg sheet were pressureless-sintered at each temperature for 1h in the furnace (in air). For the purpose of lowering sintering temperature to avoid the degradation of fibers contained green body during sintering, the $SiO_2$-$B_2O_3$-based glass powder was added to alumina powder. The weight ratio of added glass powder to alumina powder which is called alumina/glass weight ratio was varied for the optimum sintering temperature. Three-point bending test on the smooth flat specimen and single edge cracked fracture toughness test were performed at constant cross-head speed of 0.5mm/min, in order to evaluate the strength properties and fracture toughness of Si-Zr-C-O/$Al_2O_3$ composites. The sintered density of the Si-Zr-C-O/$Al_2O_3$ composites was measured by the Archimedes method. The cross-section of Si-Zr-C-O/$Al_2O_3$ composites was observed by Digital Microscope (Keyence VHX, Japan) and SEM (Quanta200, FEI, USA) before and after bending test.

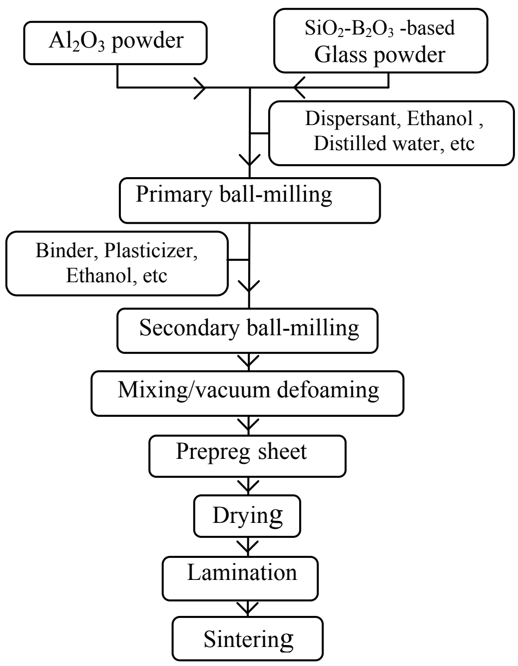


Fig.1 Fabrication process of Si-Zr-C-O/$Al_2O_3$ composite.

## 3 Experimental Results and Discussion

### 3.1 Sintered Density

Table 1 shows the sintered density of Si-Zr-C-O/$Al_2O_3$ composites which were sintered at different temperature in each alumina/glass weight ratio. Significant density reductions were observed in the specimens sintered at 830, 850 ºC in 5/5 and 920 ºC in 6/4. The density reduction of 5/5 occurred at lower temperature than 6/4. The 7/3 formed dense matrices and then density reduction was not confirmed up to 1000 ºC. Fig.2 shows the sectional micrograph of Si-Zr-C-O/$Al_2O_3$ specimen sintered at 920 ºC in 6/4. The specimen included a large number of spherical pores in the matrix. This is due to the fact that bloating, characterized by spherical pores in micrograph, takes place at the specimens [4]. The specimens in which bloating occurred swelled significantly in the laminated direction. In contrast, the Si-Zr-C-O/$Al_2O_3$ specimen sintered at 900 ºC in 7/3 achieved dense matrix as shown in Fig.3.

Table 1. Sintered density of Si-Zr-C-O/$Al_2O_3$ composites sintered at different temperature in each alumina/glass weight ratio.

| Alumina/glass weight ratio | 5/5 | 6/4 | 7/3 |
| --- | --- | --- | --- |
| Sintering temperature (ºC) | 800 830 850 | 850 880 900 920 | 900 920 950 1000 |
| Sintered density ($g/cm^3$) | 2.50 1.59 159 | 2.23 2.16 2.02 1.64 | 2.63 2.63 2.71 2.62 |

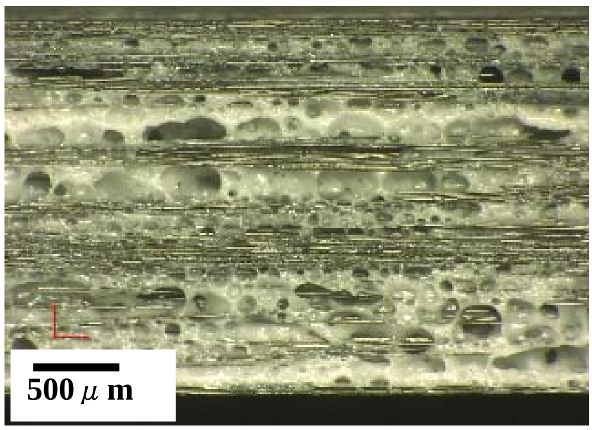


Fig.2 Sectional micrograph of Si-Zr-C-O/$Al_2O_3$ specimens sintered at 920 ℃ in 6/4.

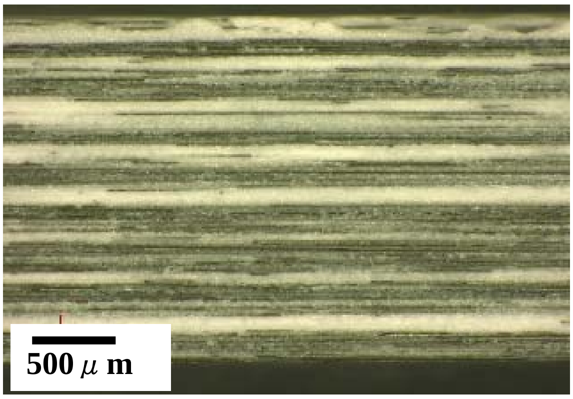


Fig.3 Sectional micrograph of Si-Zr-C-O/$Al_2O_3$ specimens sintered at 900 ℃ in 7/3.

It has been reported that a balance in competitive kinetic process between the viscous flow and dissolution of alumina into glass is required to achieve a high densification in the alumina/glass system. It should be pointed out based upon the present investigation that glass softening point, glass composition, glass ratio and particle size play an important role to achieve densification of Si-Zr-C-O/$Al_2O_3$ composites.

### 3.2 Mechanical Properties

Fig.4 shows the influence of sintering temperature and alumina/glass weight ratio on the bending strength. The highest bending strength was obtained in the specimen sintered at 900 ºC in 6/4. It should be noted that the bending strength of 6/4 remarkably increased between 850 ºC and 900 ºC though the sintered density decreased. In addition, the bending strength of 7/3 drastically reduced from 920 ºC to 950 ºC despite of similar sintered densities.
The above results indicate that the bending strength of Si-Zr-C-O/$Al_2O_3$ composites does not necessarily correspond to the sintered density and thus other parameters dominate the bending strength.
Fig.5 shows the influence of sintering temperature and alumina/glass weight ratio on the fracture toughness $K_{IC}$. The highest fracture toughness was obtained in the specimen sintered at 900 ºC in 6/4. The specimens sintered between 880 ºC and 920 ºC in 6/4 only display the high fracture toughness above 10 MPa·$m^{1/2}$, while the specimens in 5/5 and 7/3 were low fracture toughness in comparison to 6/4. It is concluded that the high bending strength and fracture toughness of Si-Zr-C-O/$Al_2O_3$ composite has been brought about by an adequate fiber/matrix interface characteristic.

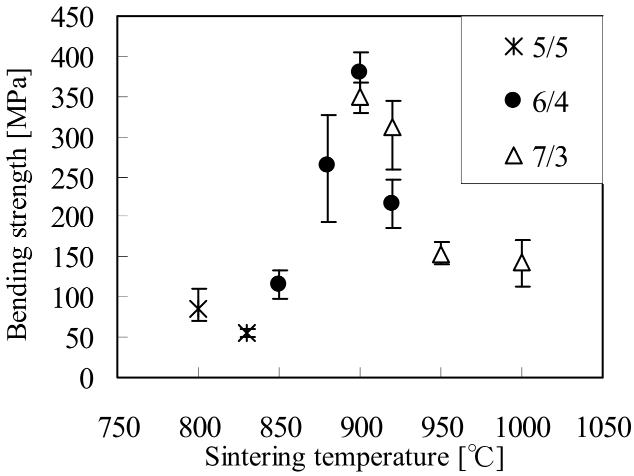


Fig.4 Comparison of the bending strength for Si-Zr-C-O/$Al_2O_3$ specimens sintered at different temperature in each alumina/glass weight ratio

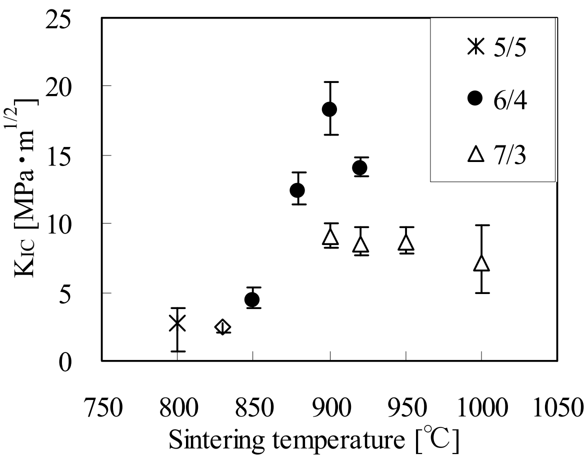


Fig.5 Comparison of the fracture toughness for Si-Zr-C-O/$Al_2O_3$ specimens sintered at different temperature in each alumina/glass weight ratio.

Fig.6 shows bending stress-deflection curves for the specimens sintered at 900 ºC in 6/4 and 7/3. The 6/4 deflected twice as much as the 7/3 and showed higher ultimate stress than the 7/3. Furthermore non-linear fracture behavior was obviously observed in 6/4.

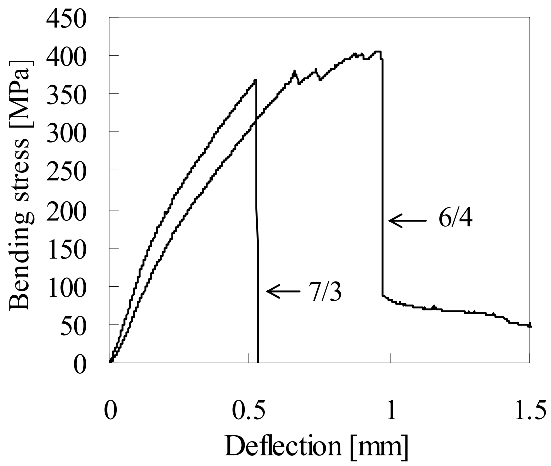


Fig.6 Bending stress-deflection curves for Si-Zr-C-O/$Al_2O_3$ specimens sintered at 900 ºC in 6/4 and 7/3.

Fig.7 and Fig.8 indicates SEM micrographs of the bending fracture surface of Si-Zr-C-O/$Al_2O_3$ specimen sintered at 900 ºC in 6/4 and 7/3. The 6/4 exhibited extensive fiber pull-out during failure, which was approximately 0.5-1.0 mm. On the other hand, the pull-out length of 7/3 was short and the fibers were decoupled. It is considered that the addition of glass powder into $Al_2O_3$ matrix plays an extremely important role to create adequate fiber/matrix interfaces and thus Si-Zr-C-O/$Al_2O_3$ composite achieves the high strength and toughness.

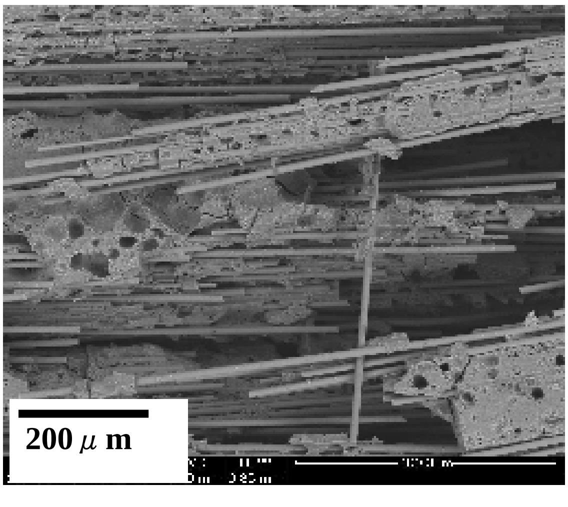


Fig.7 SEM micrograph of the fracture surface of Si-Zr-C-O/$Al_2O_3$ specimen sintered at 900 ºC in 6/4.

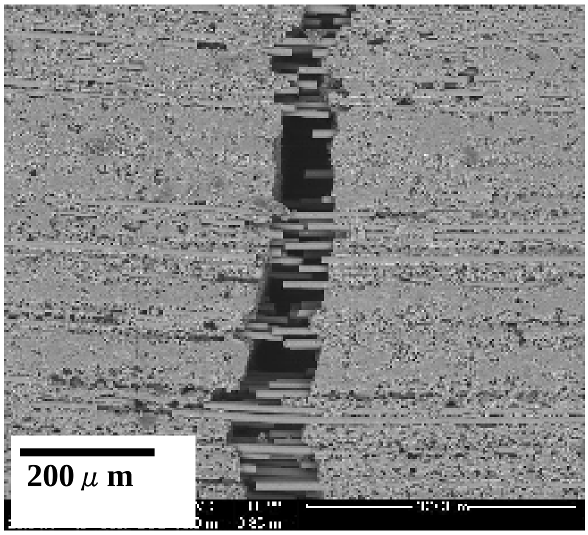


Fig.8 SEM micrograph of the fracture surface of Si-Zr-C-O/$Al_2O_3$ specimen sintered at 900 ºC in 7/3.

## 4 Conclusion

The influence of alumina/glass weight ratio, sintering temperature and sintered density affecting bending strength and fracture toughness has been investigated in the present work. The Si-Zr-C-O long fiber /$Al_2O_3$ composite sintered at 900 ºC in alumina/glass weight ratio 6/4 showed maximum bending strength of 405 MPa and fracture toughness of 20 MPa·$m^{1/2}$. It should be emphasized in conclusion that adding the glass powder to the alumina matrix play an important role not only to lower the sintering temperature, but also to provide an adequate fiber/matrix interfacial characteristic.

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