

# EFFECT OF THE FIBER/MATRIX INTERFACE ON THE IN SITU FIBER PERFORMANCE OF PIP-SiC/SiC COMPOSITE

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## ABSTRACT

To obtain an useful knowledge for the fiber/matrix interface layer design of the PIP-SiC/SiC composite, the effect of the interface layer on the tensile property was statistically characterized by tensile test, SEM observation, and Weibull analysis. The evaluations were performed for the samples of mono-filament, fiber bundle, and bundle composite of various interface layer conditions.

## 1. INTRODUCTION

SiC continuous fiber reinforced SiC matrix (SiC/SiC) composite has reliable mechanical property and excellent environmental resistance, so that it is expected to be applied to the structural material in the fields of aerospace, nuclear power generator etc. Of course, the fiber/matrix interface layer is given by chemical vapour deposition or infiltration (CVD or CVI) method. In addition, the SiC matrix is fabricated by polymer impregnation and pyrolysis (PIP) or CVD/CVI method. Both the producing process of SiC/SiC composite require the heat treatment at high temperature of roughly 1000°C. Thus, the fiber selected as the reinforcement of SiC/SiC composite is demanded to exhibit an excellent heat resistance.

However, the strength of the SiC fiber embedded in the composite has been reported to be noticeably lowered due to the damage in handling and processing [1]. It is also recently pointed out that the surrounding conditions, i.e. interface and matrix, offer big influences on the in situ fiber strength [2].

The objective of this study was to evaluate the effects of the interface layer conditions on the composite tensile performance by tensile test, SEM observation, and Weibull analysis. The evaluation was conducted for the samples of mono-filament, fiber bundle, and bundle composite of various interface layer conditions.

## 2. EXPERIMENTS

Tyranno ZMI fibers (filament diameter = 11  $\mu$  m, 800 filaments/bundle, UBE Industries,

Ltd., Japan) with non-coating, the PyC coating of various thicknesses and the multiple coating of PyC + SiC were used for the samples of filaments, fiber bundles, and bundle composites. And allylhydridepolycarbosilane (AHPCS, Starfire Systems Inc., USA) was adopted as the precursor for the matrix. For the filler material of matrix, SiC fine powder, ultra fine grade of  $\beta$ -random<sup>TM</sup> (Ibiden Co., Ltd., Japan) was used. Its average particle size was 270 nm.

Fabrication procedures for the composite samples were as follows; 1) fiber bundle was fixed to the carbon fixture, 2) C layer of 300nm, 1000nm or 3000nm thickness with/without SiC layer of 100 nm thickness was formed on the fibers by CVI method, 3) polymeric slurry mixed 70 wt% precursor polymer with 30 wt% SiC powder was impregnated into the bundle, 4) the prepreg bundle was heated at 1000°C during 1 hour in vacuum with organic gas evolutions, 5) further 7 times PIP process, which were precursor polymer impregnation and pyrolysis at 1000°C for 1h in argon atmosphere, was conducted, 6) finally, heat treatment at 1000°C for 1h in vacuum was performed so that the gas component was entirely vaporized.

The source gas in order to yield C layer and SiC layer were methane (CH<sub>4</sub>) and methyl trichlorosilane (CH<sub>3</sub>SiCl<sub>3</sub>), respectively. Through the sequential process, linear composite samples of more than 150 mm length and around 1mm diameter were obtained.

The tensile test was conducted under displacement rates of 0.1mm/min for filament samples and 0.5 mm/min for bundle and composite samples, respectively, at room temperature. The gage length was 90 mm. At least 5 samples were tested for each fabrication condition. In order to reduce the error due to stress concentrations from the clamp, the data of fracture within 5 mm distance from each end of the gage part were removed. After the tensile tests, fiber fracture surfaces were observed by SEM in order to measure the fiber diameter because tyranno ZMI fiber has wide distribution of diameter and to check the fracture condition. Theoretical strengths of the bundles were calculated by using the Colemann's equations [3].

### **3. RESULTS AND DISCUSSION**

In the following description, it was assumed that the tensile load had been applied to only the fiber, not to interfacial layer and matrix, because the sample of SiC/SiC composite without interfacial layer could not have been fabricated maybe due to its negligible low strength.

Fig.1 showed the maximum tensile loads of various carbon layer thickness samples. Firstly, the SiC/SiC composite with C layer of 300nm thickness exhibit the best tensile

performance in these conditions. And no difference was recognized between 1000nm and 3000nm. This tendency corresponds with Curtin's theory [4]. Secondary, the tensile maximum load of the composite with C and SiC layer was higher than that of the composite with only C layer. Further, the distribution of maximum load of SiC coated samples was wider than that of samples without SiC layer. This suggests that the SiC coating over C layer is not good in focus of the improvement of composite mechanical property, although the SiC coating is expected to barrier the C coating against oxidative attack from matrix crack. Also, the maximum load distribution of the bundle sample with C and SiC layer was not wide. Thus the composite distribution becomes wider when the fiber, C and SiC layer, and SiC matrix get together.

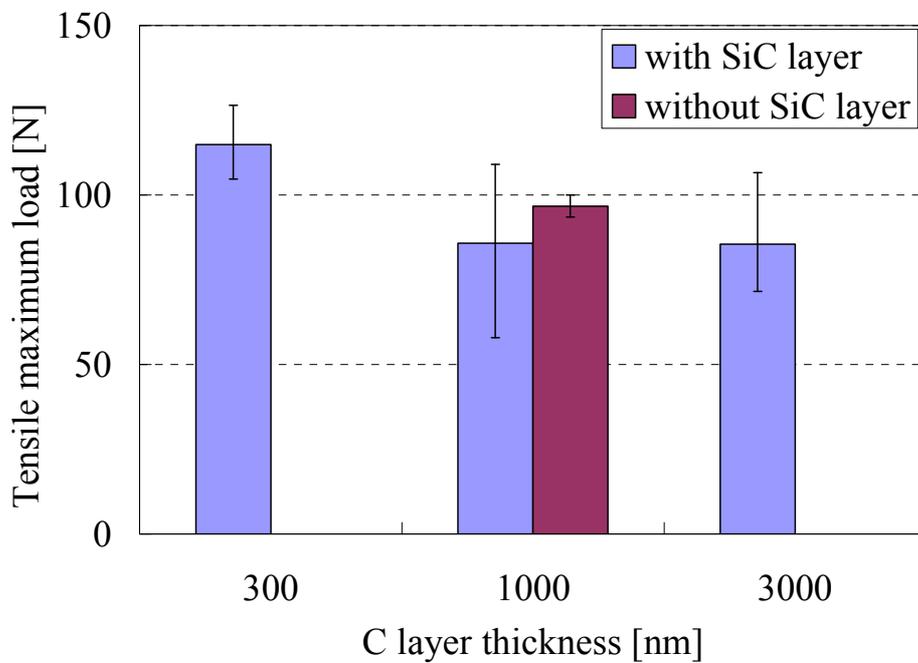


Figure 1: The maximum tensile loads of SiC/SiC bundle composites with various interfacial layers.

Fig.2 showed the tensile strength for the filament samples of various conditions. The strength was estimated from the maximum load of the result of tensile test and the filament diameter measured by SEM observation of fracture surface of the filament sample. Further, the data was applied to two parameter Weibull analysis using the two equations followed by

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad (1)$$

$$\sigma_m = \sigma_0 \Gamma\left(1 + \frac{1}{m}\right) \quad (2)$$

where  $F(\sigma)$  is the cumulative fracture probability,  $\sigma_0$  is scale parameter,  $m$  is shape parameter, and  $\sigma_m$  is the mean strength in the Weibull distribution. From the result of the analysis, the strengths of filament samples with coating layers noticeably decreased compared to filament sample which was not heated. In order to investigate the effect of heat treatment in the CVI and PIP process, filament sample heated at 1100°C for 15h in vacuum assumed to be about the same heat treatment of CVI process was tested. The result of this, heated sample markedly deteriorated as well as coated samples. Thus, the coated filament sample was thermally damaged in the CVI process. And tyranno ZMI fiber does not seem to satisfy the demand of heat resistance against the heat treatment in CVI process. So it will be important to find out the factor of increasing the distribution range of the composite strength.

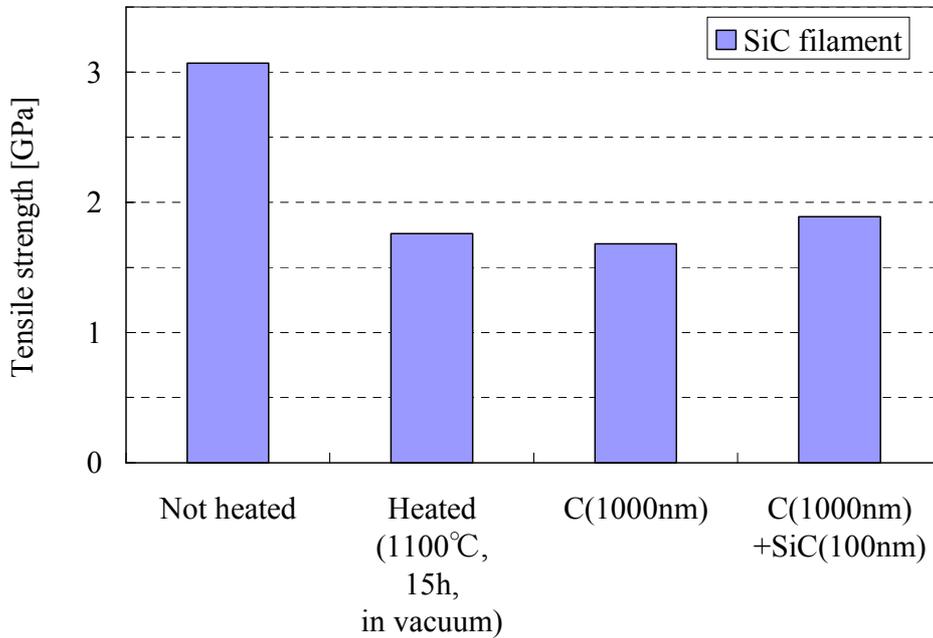


Figure 2: The tensile strengths of filament samples of various conditions.

The strengths of bundle and composite specimens were calculated by using the maximum load from the result of tensile test and the catalog value of 11  $\mu$  m of filament

diameter and 800 filaments per bundle. In order to estimate the theoretical bundle strength, the filament strength was applied to Coleman's equation [3] followed by

$$\sigma_{th} = \sigma_m \left( \frac{1}{me} \right)^{1/m} \frac{1}{\Gamma(1+1/m)} \quad (3)$$

where  $\sigma_{th}$  is the theoretical bundle strength. Fig.3 shows the tensile strength of various specimens of various conditions. Here, it was the bundle that was the basic constituent of the composite from a practical application standpoint, so that the strengths were compared on the basis of the bundle. The comparison between theoretical and practical bundle strength represents. In Fig.3, the practical bundle strength was much lower than theoretical bundle strength, thus the effect of the contact and sliding between filaments in practical bundle as shown in Fig.4 offer big influence on the practical bundle strength. And also, the composite strength was much higher than the practical bundle strength. This may be why the matrix composite effect occurs due to the addition of matrix. Thus, the composite effect offers the big influence on the practical bundle strength. By the way, these are macroscopic estimation. In the future, it will be necessary to evaluate microscopic estimation of mirror radii measurements and push-out tests in order to estimate the in situ strength of fiber in SiC/SiC composite.

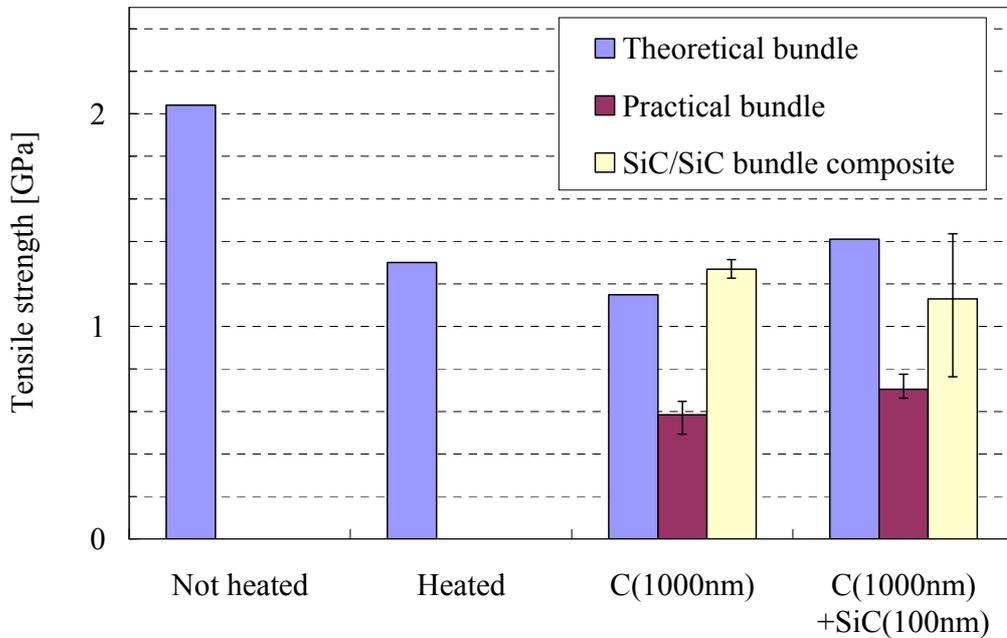


Figure 3: Comparison of the tensile strength for various sample on the basis of the bundle.

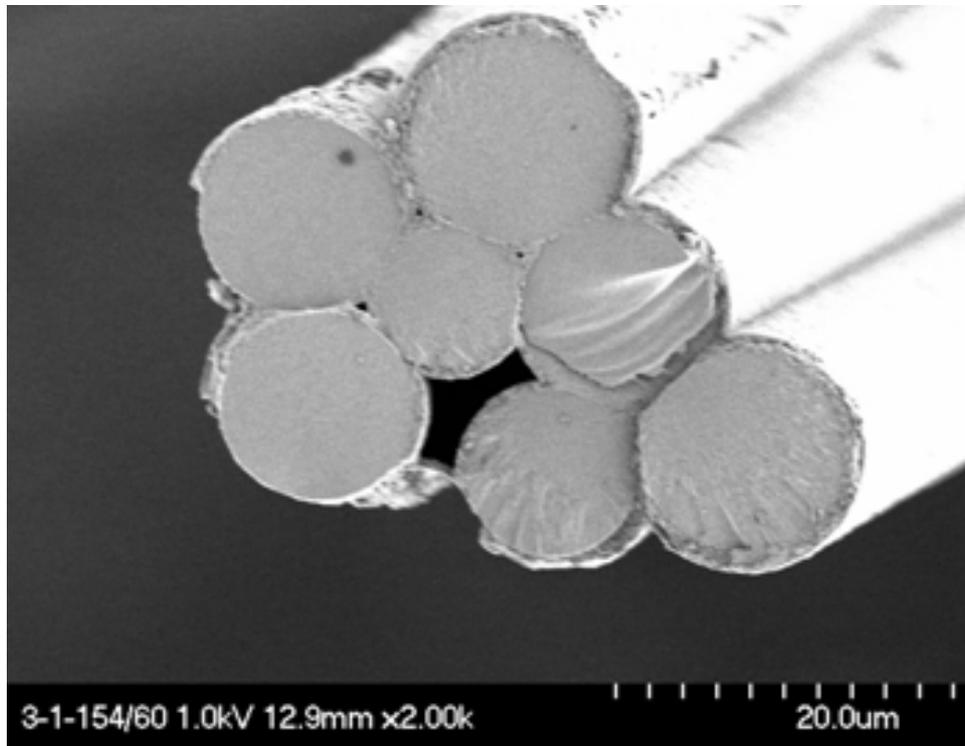


Figure 4: A SEM micrograph of the fracture surface for SiC bundle coated with 3000nm thick C and 100nm thick SiC layer.

#### 4. SUMMARY

- 1) The comparatively thin sample has exhibited better tensile property for the SiC/SiC bundle composite with various thick C and 100nm thick SiC interfacial layer. And also, there didn't seem to be effect of the C layer thickness on the tensile property in excess of a certain thickness. It was indicated that the possibility of the strength degradation and increasing its distribution by the SiC overcoating on the C layer.
- 2) Tyranno ZMI fiber has been assumed to be able to use without remarkable strength degradation to 1300°C in Ar, but indeed the heat treatment at 1100°C for 15h in vacuum supposed to be about the same heat treatment in the CVI and PIP process deteriorated the filament strength more than about 40% of its original strength.
- 3) Comparison between the theoretical and practical bundle strength showed that the effect of the contact and sliding between filaments in practical bundle offer big influence on the practical bundle strength. And also, comparison between the practical bundle and bundle composite strength showed that the effect of the addition of matrix to interfacial layer coated bundle offer big influence on the

practical bundle strength.

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