

# THE MICROSTRUCTURE CONTROLLING OF CARBON FIBER IN C/SiC COMPOSITES

Zou Wu, Yang Lianshen, Song Maili, Wang Tao  
Shaanxi Non-metallic and Technology Institute  
P.O.Box 101 Xi'An ,Shaanxi 710025 P.R.China

## Introduction

Carbon fiber reinforced silicon carbide matrix composites have a potential application in high temperature thermal structure of aerospace vehicles and motor propulsion systems. The workhorse manufacturing methods of the C/SiC composites includes polymer precursor impregnation & pyrolysis (PIP) and chemical vapor infiltration (CVI), whose manufacturing periods are very long. So we developed the CVI+PIP combined process [1]:

CVI+ PIP

C<sub>f</sub> Weaving Preform → C/SiC Composites

In this paper, the effects of pretreatment, coating, densifying, and stabilization process factors on the microstructure of the carbon fibers were investigated, in order to raise the fiber residual strength and the composite properties.

## Experiment

HS-1 carbon fiber was used in the experiment. Phonic resin, pitch, polycarbiline, and propylene were used as coating precursor. The carbon fibers passed through four stages: (1) pretreatment at 900 °C in N<sub>2</sub>; (2) Coating treatment by precursor solution soaking or chemical vapor deposition (CVD) at 900 °C in N<sub>2</sub>; (3) densifying treatment by CVI+PIP at 900 °C-1200 °C; (4) stabilizing treatment at 1500 °C in N<sub>2</sub>.

In the C/SiC composites, the fiber content is about 50%. The preforms were densified first to 1.70g/cm<sup>3</sup> by CVI from CH<sub>3</sub>SiCl<sub>3</sub>-H<sub>2</sub> system, then from 1.70g/cm<sup>3</sup> to more than 1.9 g/cm<sup>3</sup> by PIP from precursor solution. The fiber tensile properties and the composite flexural strength and

fracture toughness were tested. The microstructure of carbon fibers and the composite fracture surface were analyzed through SEM.

## Results and Discussion

HS-1 carbon fiber is protected by epoxy sizing agent with the content of 0.7 percent. When heat treated at 1500 °C without protection coating, not only the fiber defects (such as grooves, dents and cracks, showed as figure 1) were exposed and increased because of the atmosphere erosion and the non-carbon substances volatilizing, but also the crystal grains grew. That caused 32% strength loss, 20% modulus increment, and 46% elongation-to-break loss of the carbon fibers, described as table 1.

Showed as figure 2, coating the carbon fibers with pyrolytic carbon can remedy and reduce the fiber defects, provide pulling tension to increase the fiber crystal orientation to axial during the later heat treatment, and protect the carbon fibers from being corroded by the exhaust gas atmosphere. That resulted in the fiber residual strength rising by 28.7%, showed as table 2.

Further, the fiber residual strength of the CVD-carbon coating fibers is not the highest among the studied kinds of pyrolytic carbon coatings, but the flexural strength and fracture toughness of its composites with 0.7 μm thick coating came to 436Mpa and 13.1Mpa.m<sup>1/2</sup> and were obviously higher than of the others. For better interface bond was achieved in the C/SiC composites, showed as figure 3.

## Conclusions

Heat treatment will not only expose and increase the

fiber defects but also cause the crystal grains growing. CVD-carbon coatings can raise the fiber residual strength because of its remedying and reducing the fiber defects and enhancing the fiber crystal orientation to axial. Along with its adjusting the bond between carbon fiber and silicon carbide matrix, the flexural strength and fracture toughness of the C/SiC composites reinforced by the coated fibers were obviously improved.

## Reference

1. Zou Wu, etc. Triaxially Braided C/SiC Composites: Research & Development by the Combined Process of CVI+PIP, Encera'98 & HT-CMC 3 (Osaka, Japan); Japanese Ceramic Society, 1998; 153—157

Table 1. The single filament tensile properties of HS-1 carbon fibers after heat treatment

Heat Treatment	Uncoated	1500°C, N <sub>2</sub>	2500°C, N <sub>2</sub>
Tensile Strength, MPa; (cv%)	3290	2240 (22.5%)	2130 (23.9%)
Tensile Modulus, GPa; (cv%)	225	270 (11.0%)	375 (8.5%)
Elongation-to-break, %; (cv%)	1.49	0.81 (20.0%)	0.57 (22.6%)
D <sub>002</sub> , nm	0.3531	-----	0.3406
Lc, nm	1.5	-----	14.9
Graphite Content, %	-----	-----	39

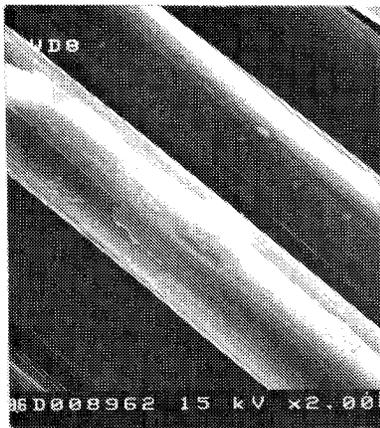


Figure 1. The uncoated fibers with defects

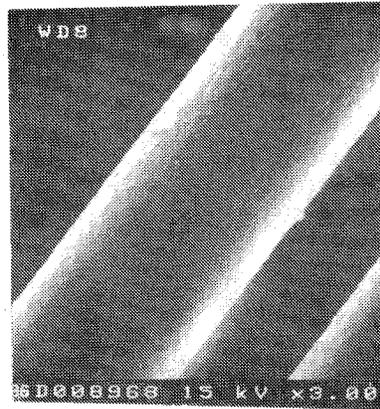


Figure 2. The coated fibers

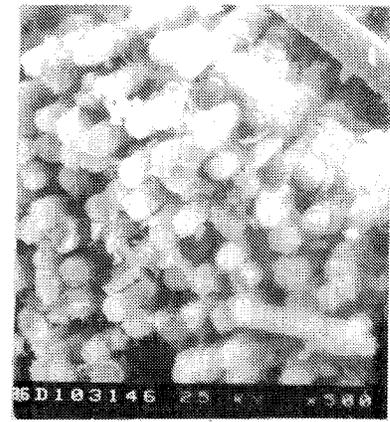


Figure 3. The composite fracture surface

Table 2. The properties of the coated fibers and the C/SiC composites

Precursor	Coating Process	Fiber Tensile Strength (MPa)	Flexural Strength* (MPa)	Fracture Toughness* (MPa.m <sup>1/2</sup> )
-----	uncoated	2241	165.1	5.7
Phonetic	solution soaking	2431	204.6	-----
Pitch	solution soaking	2340	294.7	7.4
Pitch+polycarbiline	solution soaking	2883	221.7	-----
Propylene	CVD	2730	436.2	17.9

Table 3. The effects of the CVD-carbon coating thickness on the fiber properties

Coating Thickness (μm)	Tensile Strength (GPa)	Tensile Modulus (GPa)	Elongation-to-break (%)
Uncoated	1.22	190.9	0.66
0.32	1.64	203.5	0.85
0.48	1.68	206.7	0.85
0.74	2.65	209.4	1.32