Microstructure Evolution of Carbon Fiber in PIP-processed C/SiC Composites under High Temperature Environment

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***Abstract*—The thermal stability of carbon fiber in PIP-processed C/SiC composites under high temperature environment was studied. The microstructure evolution of carbon fiber in PIP-C/SiC composites was characterized. The results showed that the microstructure of carbon fiber gradually destroyed with the elevation of annealing temperature. When the annealing temperature reached 1800℃, the integrity of carbon fiber was destroyed, which corresponding to the descend of mechanical properties. Further study on the mechanisms of carbon fiber is needed and is in progress.**

Keywords-C/SiC composites; PIP; carbon fiber; high temperature

I. INTRODUCTION

The incorporation of continuous carbon fibers into ceramics offered a promising route to the production of tough and reliable ceramic materials [1]. Due to their low specific weight, high strength, excellent resistance to ablation as well as cost effective production, carbon fiber reinforced silicon carbide (C/SiC) composites represented an interesting material for high-temperature structural applications [2-4].

The most common techniques for producing C/SiC composites were Chemical Vapor Infiltration (CVI), Liquid Silicon Infiltration (LSI) and Polymer Infiltration and Pyrolysis (PIP), or a combination of the above. PIP process was conversely much cost and time effective [5-10].

In PIP process, a critical issue for utilization of carbon fibers was their thermal stability. The carbon fibers were vulnerable to severe property degradation due to decomposition, oxidation, accelerated grain growth, or a combination of the above during high-temperature processing and service environment [11].

The purpose of the present paper was to evaluate the microstructure evolution of carbon fiber in PIP-processed C/SiC composites under high temperature environment is discussed.

II. EXPERIMENTAL PROCEDINGS

A. Morphology Preparation of PIP-C/SiC composites

Polycarbosilane (PCS) (molecular weight: 1742, soften point: 175℃) was synthesized in our laboratory. Xylene was used as the solvent for PCS.

Three-dimensional braided carbon fibers (T-300, ex-PAN carbon fibers, Toray) were used as the reinforcement, and the fibers volume fraction was 45%. 9~12 cycles of infiltration of PCS-Xylene solution (mass ratio1: 1) and subsequently pyrolyzed at 1200℃ under N_2 (purity: 99.99%) atmosphere [3].

B. High-temperature annealing experiment and Samples characterization

High temperature annealing processes were carried out by annealing between 1400℃ and 2000℃ for 1h under Ar atmosphere.

The microstructures of the samples after high-temperature annealing experiment were examined by scanning electron microscopy (SEM, FEI Quanta-200).

III. RESULTS AND DISCUSSION

A. Morphology of carbon fiber in PIP-processed C/SiC composites

The typical morphology of the carbon fiber is shown in Figure 1. The fiber is near rotundity in shape and the diameter is about 7μm.

The surface of the fiber is rough. It can be seen that there are visible cracks on the surface, which are caused by PIP process. PCS shrinks in pyrolysis, and then O element in the PCS is reacted with carbon fiber, which may result cracks on the surface of the fibers.

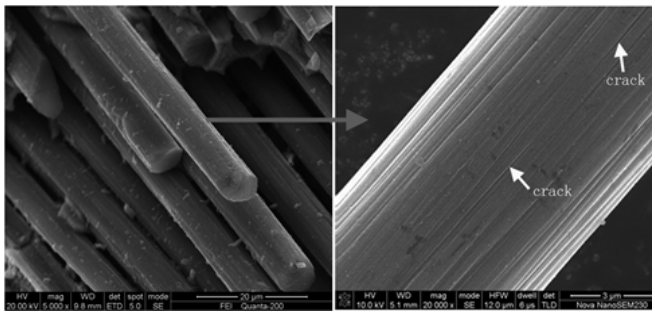

Figure 1. Surface morphology of as-fabricated carbon fibers

It was well known that the interfacial bonding strength had a great effect on the mechanical properties of C/SiC composites [12]. When the fibers were coated by SiC matrix, it reinforces the interfacial bonding and improved the accommodation of the fiber/matrix. As a result, the composites exhibited higher flexural strength and an evident toughened fracture behavior [4].

B. Microstructure evolution of carbon fiber in PIP-processed C/SiC composites

The microstructure of carbon fiber in PIP-processed C/SiC composites under high temperature environment is shown in Figure 2-Figure 5. The changes of carbon fiber after high temperature annealing processes are verified by the SEM.

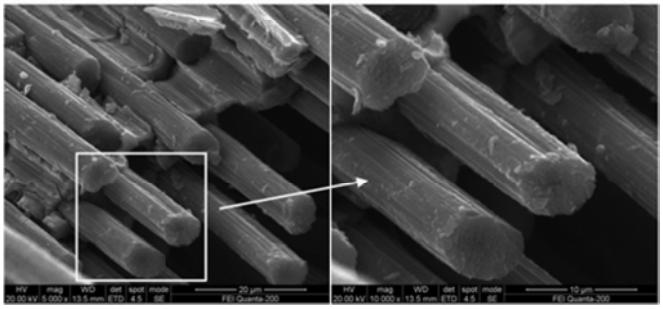

Figure 2. Surface morphology of carbon fibers under 1400℃ annealing

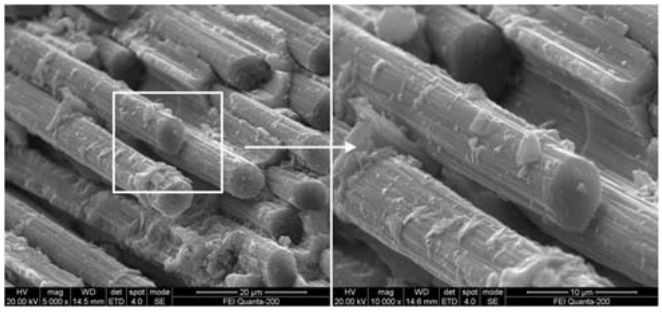

Figure 3. Surface morphology of carbon fibers under 1600℃ annealing

Compared with the similarly visible difference in surface morphology under different temperature, there is distinct surface morphologic change between the samples shown in Figure 2-Figure 5. Together with above-mentioned features, a very interesting phenomenon is also observed. The reasons of mechanical behavior of PIP-processed C/SiC composites [13] can be understood from inspection of SEM images (Figure 2-Figure 5).

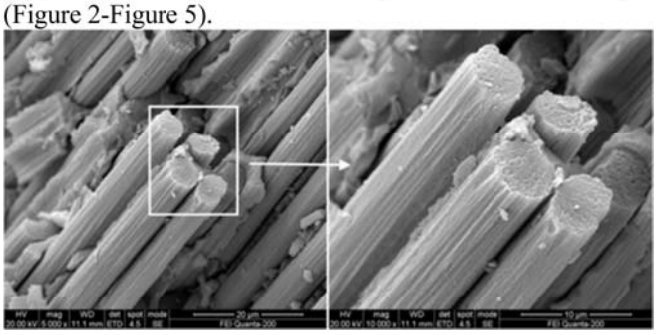

Figure 4. Surface morphology of carbon fibers under 1800℃ annealing

The SEM images of carbon fibers after treatment at 1400℃ are presented in Figure 2. One can observe large cracks in the fibers.

The formation of large size cracks can be connected with coalescence of small size cracks and diffusion of them outward. The version on the formation of cracks due to the interaction of carbon fibers and PCS is unlikely because of high-temperature in the process. One can also propose that cracks could originate from gas evolution (e.g. hydrogen) from carbon fibers themselves during the high-temperature treatment.

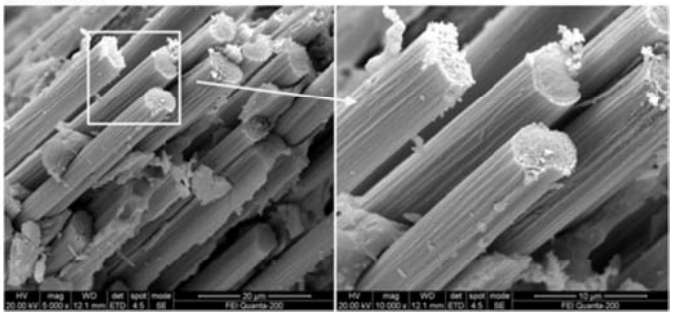

Figure 5. Surface morphology of carbon fibers under 2000℃ annealing

The SEM images of carbon fibers after treatment at 1600℃ are presented in Figure 3. For the fracture of carbon fibers, the CF is not clean and the matrix is not comparatively neat, which indicated a strong interface. With the treatment, the matrix became rougher and more SiC matrix attached to CF. An amount of resin adhering to the carbon fiber surface could be seen (marked in circles), which is an evidence of a stronger interphase. At the fiber surface with many large catalyst particles (bright contrast in the SEM image) the pitting is significantly deeper and densely distributed. This observation indicates that excessive etching of the carbon fiber surface occurred at large catalyst particles, as will be explained later in the text.

One can see that with increasing temperature to 1800℃, the morphology of the carbon fibers is drastically changed (Figure 4). Due to an agglomeration and recrystallization of carbon particles, the smooth areas are observed, whereas the other areas become loose.

The carbon fibers cannot retain their morphology after high-temperature treatment, which corresponding the decline

of mechanical behavior of PIP-processed C/SiC composites [13].

The carbon fibers under 2000℃ annealing are thinner compared with the others (Figure 5), the microstructure of the carbon fibers sharply changes. The integrity of carbon fiber is destroyed, which homologous to the tragedy of mechanical properties [13].

Therefore, the microstructure of carbon fibers sharply changes after high-temperature heat treatment.

Further study on the mechanisms of carbon fiber is needed and is in progress.

IV. CONCLUSIONS

The thermal stability of carbon fiber in PIP-processed C/SiC composites under high temperature environment was studied:

- The microstructure of carbon fiber gradually destroyed with the elevation of annealing temperature.
- When the annealing temperature reached 1800℃, the integrity of carbon fiber was destroyed, which corresponding to the descend of mechanical properties.
- Further study on the mechanisms of carbon fiber is needed and is in progress.

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