OXIDATION OF CARBON FIBER AND ITS EFFECT ON MECHANICAL PROPERTIES

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Abstract

Carbon fibers are the main component and load carrier in many high-temperature composites. Oxidative ablation of carbon fibers is the main failure mechanism for high-temperature composites and seriously affects their mechanical properties. In this study, the oxidative ablation behavior of carbon fibers from room temperature to 900 °C was systematically investigated using a thermogravimetric analyzer and in situ video detection. Carbon fiber weight loss occurred above 600 °C in air. The video images showed that the carbon fiber surfaces changed color significantly, and then the fibers gradually disappeared. This shows that the carbon fibers underwent a chemical reaction, i.e., oxidative ablation. The oxidative ablation behavior of carbon fibers was also investigated by determining the strength of the carbon fibers at specific temperatures. The carbon fiber strength decreased above 400 °C.

1. Introduction

Rapid developments in the aerospace industry have resulted in increasing demand for carbon-fiber-reinforced composite materials [1]. Because of their low density, high strength, low coefficient of thermal expansion, and other good properties [2-3], carbon fibers are often used as the main bearing unit in composites. The service temperatures of composite materials are generally high, therefore oxidative ablation is the main reason for the failure of carbon fibers and the mechanical properties of composites. This leads to problems regarding the reliability of composite materials during use [4]. It is therefore important to study the oxidative ablation behavior of carbon fibers and the effects of ablation on their mechanical properties to enable improvements in the reliability of carbon fiber composites at high temperatures. At present time, the numerical simulation about composite oxidation has been performed [5], but the study of fiber oxidation and its influence on mechanical behavior is not well studied. There are few reports in the literature of studies of carbon fiber oxidative ablation behavior and its effects on mechanical properties at different temperatures, and the available data vary greatly. For example, Eckstein[6] reported that carbon fiber oxidation begins at 230 °C, but Srivastava[7] found that carbon fiber oxidation did not start until 350 °C. Tanabe et al.developed a model based on the mass loss in the oxidation process and used it to characterize the oxidative ablation behavior of carbon fibers at different temperatures quantitatively. However, these studies lack direct observations of ablation during carbon fiber oxidation, and do not give an intuitive understanding of the ablation process and its relationship with mechanical properties. In this work, we used thermogravimetric analysis (TGA) and a laboratory-developed high-temperature video on-line observation instrument to study carbon fibers. The ablation behavior of the carbon fibers and their mechanical properties at different temperatures and in different ablation phases were studied systematically.

2. Materials and methods

2.1. TGA

a. The sample (about 4.5 mg) was placed in a 70 μL ceramic crucible. Air was introduced at a rate of 40 mL/min to ensure a sufficient oxygen concentration. TGA was performed using a Mettler Toledo TGA/DSC 1 1600HT instrument. We conducted a blank experiment under the same conditions for comparison to eliminate interference errors.

b. The temperature was increased from room temperature to 900 °C at a rate of 20 °C/min. Based on the experimental mass loss results, 300, 500, 600, and 850 °C were set as test temperature points. The temperature was raised to these points at a rate of 60 °C/min and TGA was performed at constant temperature.

2.2. In situ video observations of carbon fiber oxidative ablation

The carbon fiber morphology during oxidative ablation was observed in situ to verify the oxidative ablation behavior indicated by the TGA experiments. We performed the in situ observations using a device that we developed. The system, which is shown in Fig. 1, consists of a micro high-temperature furnace with a cavity of diameter 10 mm in the middle of the furnace and a maximum temperature of 900 °C; a charge-coupled device (CCD) camera and telephoto microscope (working distance 89 mm, maximum optical magnification 6×); a light-emitting diode as a light source; a temperature control box; and a computer for compiling the results. In the experiment, the chopped fiber was placed on the flat end of a quartz-glass rod of diameter 8 mm, and this end of the glass rod was placed in the middle of the cavity in the high-temperature furnace. A glass sheet covered the upper end of the furnace cavity during heating. The CCD was used to focus the carbon fibers and images were collected at regular intervals and stored in the computer.

2.3. Carbon fiber tensile strength measurements

2.3.1. Tensile test specimens

We used T300 fiber bundles as the experimental objects to facilitate the experiments. To ensure a uniform fiber yarn, and therefore ensure experimental accuracy, we used a national standard (GB3362) for the fiber bundle specimens. The fiber bundles were straightened and fully impregnated in an epoxy resin (BHC-166, resin: hardener weight ratio 100: 84) so that the filaments were bonded to each other, and then kept under natural drooping conditions. The resin paste was cured for 3 h in a temperature box. The two ends of the fiber bundle were pasted with kraft paper to facilitate loading and avoid slipping. The dimensions of the kraft paper were 45and 15mm, with an effective length of 150 mm.

2.3.2. Tensile test device

Tensile tests were performed using a testing machine with a measuring range of 5000 N; the stretching speed was adjustable. In the tests, the small furnace shown in Fig.1 was used to locally heat the specimen passing through its inner cavity, as shown in Fig. 2.

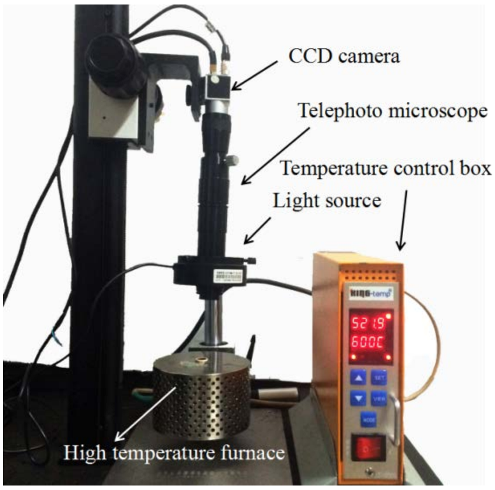


Fig.1. Fiber ablation observation device

Fig. 2. Local details of fiber bundle specimen in high-temperature tensile test (preheating stage before loading)

3. Results and discussion

3.1. TGA

The results for TGA performed from room temperature to 900 °C at a heating rate of 20 °C/min are shown in Fig. 3.The results show that oxidative ablation of the carbon fibers began at 500 °C.

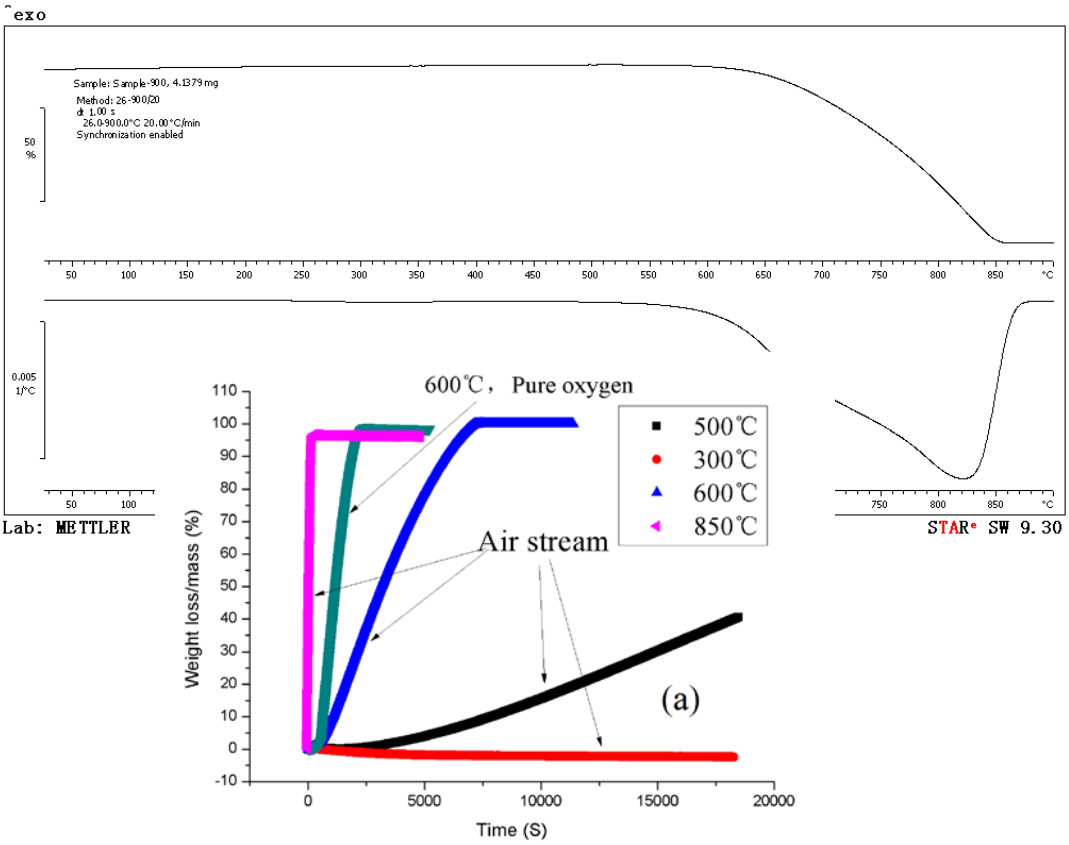


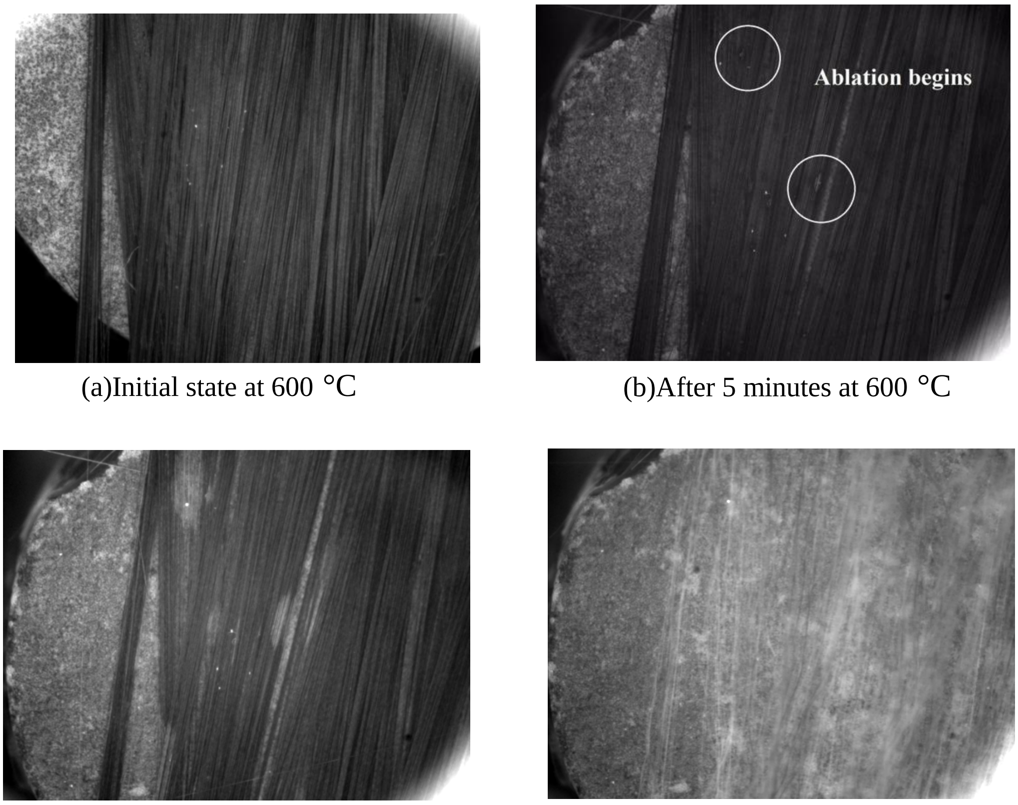
Fig. 3. TGA curves for oxidative ablation of carbon fibers (room temperature to 900 °C, heating rate 20 °C/min); upper: weight loss ratio with temperature; lower: weight loss rate with temperature

Based on these results, we performed several constant-temperature TGA experiments, i.e., at 300,500, 600, and 850 °C. The temperature was increased rapidly to the predetermined value and maintained for 1 to 5 h. The thermal weight loss rate of the carbon fibers with time was determined. The results are summarized in Fig. 4.

Fig. 4 Relationship between weight loss ratio of carbon fibers and time at different temperatures

3.2. Observation of carbon fiber oxidative ablation

We developed a video monitoring system and used it to observe the morphological changes in carbon fibers during oxidative ablation at various temperatures from 500 to 850 °C. Typical results are shown in Fig. 5.



(a)Initial state at 600 °C

(b)After 5 minutes at 600 °C

(c)After 70minutes at 600 °C

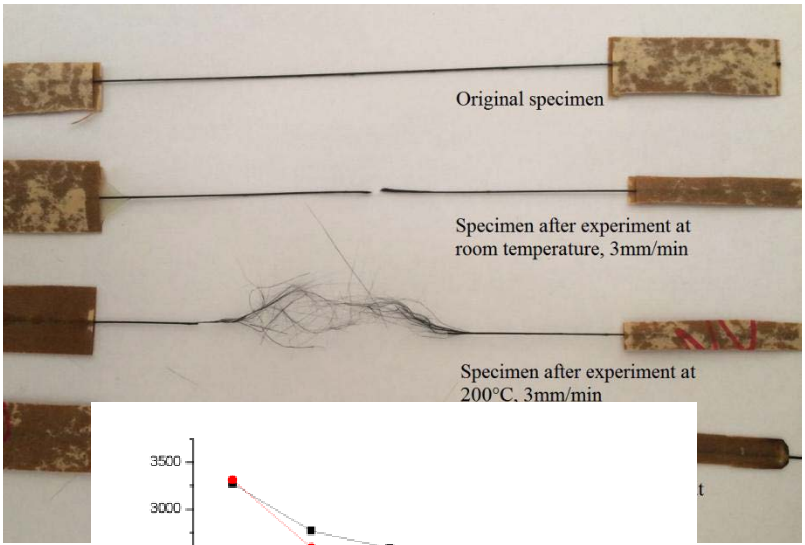
(d)After 140minutes at 600 °C

Fig. 5 Morphological changes in carbon fibers during ablation at different temperatures

Fig. 5(a)–(d) show the entire process of oxidative ablation of carbon fibers at 600 °C. At 600 °C, significant ablation had occurred after 5 min and ablation was complete after 140 min.

3.3. Tensile strength of carbon fibers at different temperatures

The mechanical properties of the carbon fibers were tested at room temperature, and 100, 200, 300, 400, 500, and 600 °C based on the carbon fiber oxidative ablation results, to guarantee the significance of the experimental results. Significant carbon fiber ablation occurred after holding the temperature at 600 °C for more than 5 min, therefore the carbon fiber mechanical properties were tested 3 and 5 min after reaching the specified temperature points. Loading rate of 3 mm/min was used. In the experiments, specimens were cut from the middle (the highest-temperature region); the fractured specimens at different temperatures are shown in Fig. 6. It was found that the epoxy gel on the fiber bundles above 200 °C was completely decomposed and volatilized, which was favorable to the electron microscopic analysis.



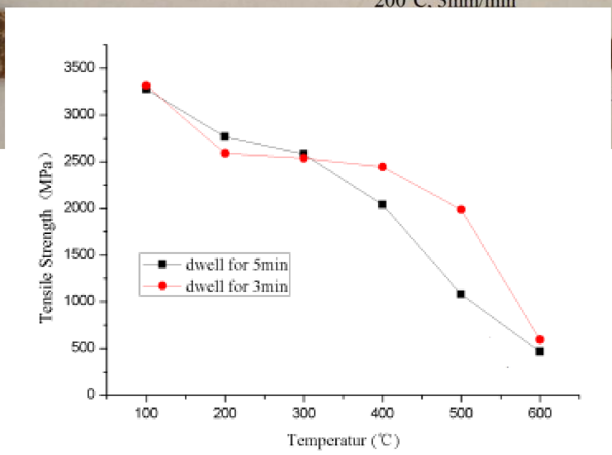


Fig. 6. Fracture of specimens at different temperatures

The tensile strength of the carbon fibers at room temperature was 3.71 GPa, which is consistent with the data provided by the supplier. The experimental results at 100 to 600 °C are summarized in Fig. 7.

Fig. 7. Tensile strengths of carbon fibers at different temperatures

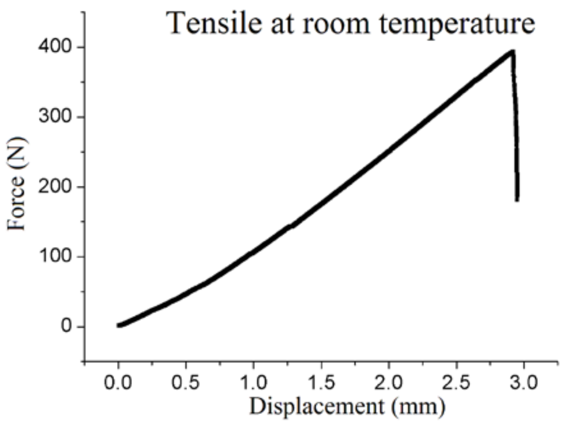


Fig. 8. Typical tensile curves for carbon fibers under different experimental conditions

It should be noted that in the mechanical property experiments the carbon fibers showed the basic characteristics of brittle fracture; a typical tensile curve is shown in Fig. 8.

4. Conclusions

In this study, the oxidative ablation behavior of T300 carbon fibers at different temperatures was systematically investigated using TGA and video on-line observations. The apparent tensile strengths of the carbon fibers from room temperature to 600 °C were tested at different temperatures. The results provide data that will be useful in performance design and failure analysis of high-temperature composites with carbon fibers as the main load cell.

The main conclusions of this study are as follows.

(1) In an air atmosphere, carbon fibers can be oxidized and ablated at 500 °C, but the rate is very slow. The oxidative ablation rate increases rapidly with increasing temperature. At 850 °C, oxidative ablation of carbon fibers is com-

plete within a few minutes.

(2) The tensile strength of the carbon fibers does not change at 300 °C. Above 500 °C, ablation causes a decrease in the nominal tensile strength or load capacity.

(3) Below 600 °C, the carbon fibers are basically brittle, and the fiber fractures are straight.

Acknowledgment

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