



Influence of thermal treatment on thermo-mechanical stability and surface composition of carbon fiber



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ABSTRACT

For investigating the influence of thermal treatment on the thermo-mechanical stability and the carbon fiber's surface composition, the T300 carbon fibers were thermally treated at temperatures ranging from 600 °C to 1500 °C. After treatment, the thermal stability and microstructure were analyzed by mass change and XRD measurement. The mechanical properties and the surface composition of carbon fibers were measured as a function of temperature by the single filament tensile test technique and ESCA analysis, respectively. The results indicated that T300 fiber had good thermal stability and high temperature mechanical properties. To some extent, the high temperature treatment could lead to a further graphitization of carbon fiber. The surface composition measured by ESCA revealed that the oxygen concentrations on the surface of fibers significantly decreased from 17.97% for as-received fiber to 1.97% for 1500 °C treated fibers. But the relative content of graphite-like carbon increased from 42.97% for as-received fibers to 53.13% for 1500 °C treated fibers. Such composition would result in a decreased surface activity of carbon fiber.

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1. Introduction

Carbon fibers offer many advantages such as light-weight and excellent thermal stability and mechanical properties at temperatures ranging from very low (liquid oxygen) to ultra-high temperature (over 1600 °C). Carbon fiber reinforced composites have been used in a number of applications such as aerospace and hi-tech products [1–5].

To utilize the excellent mechanical properties of carbon fibers in composites, it is necessary to have a desirable fiber–matrix interface to effectively transfer the load from one fiber to another through the neighboring matrix. However, the fiber–matrix interfacial properties largely depend on the carbon fiber's surface composition which affects the fiber–matrix interaction. As a result, many techniques have been applied to treat the fiber's surface in order to get a suitable interface and composites with desired properties [6–8]. In fact, the nature of the interface and its control are extremely complex.

Results in the literatures [3,4,6–8] found that the fiber–matrix interaction strongly depends upon the surface functional groups of the carbon fibers and the matrix precursor. Surface oxidation of carbon fibers is one of the main techniques, which can increase the

number of functional groups on the carbon fiber surface, and then strengthen the interfacial bonding. In particular, the mechanical properties of C/C composites are very sensitive to the bonding of interface and its load transferring capability. Also, there are some data available on the analysis of surface functional groups of carbon fibers and their influence on the mechanical properties of polymer matrix composites. A quantitative correlation between the amount of surface functional groups and the composite properties has been studied by Fitzer et al. [8]. The fabrication of C/C–SiC composites by liquid silicon infiltration technique often experiences the pyrolysis step of CFRP [1,5]. During the pyrolysis of CFRP, some experiments have found that the surface functional groups of carbon fibers is a dominant factor governing the microstructure features of C/C preform [5], which strongly affects the microstructure integrity and mechanical properties of final product, C/C–SiC composites.

On the other hand, in many cases, a strong interface bonding is not always needed for mechanical properties of composites, especially for ceramic matrix composites [1,5,9]. The pretreatment of carbon fiber has been applied in the fabrication of carbon fiber reinforced C–SiC matrix composites [5,7]. In such case, it was still not clear which properties of the carbon fibers were affected by thermal treatment and how the surface composition of fiber affected the microstructure and mechanical properties of C–SiC matrix composites.

In order to gain an insight into how the thermal treatment of carbon fibers affects the properties of carbon fiber reinforced ceramic matrix composites, in the present work, the T300 fibers

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Table 1
Physical and mechanical properties of carbon fiber.

Fiber type	T300
Filament amount	3000
Density (g/cm ³)	1.76
Average diameter (μm)	7.0
Tensile strength (GPa)	3.53
Tensile modulus (GPa)	230
Failure strain (%)	1.5

were thermally treated at elevated temperatures. After treatment, the thermo-mechanical stability and the surface composition of carbon fibers were measured as a function of temperature by the single filament tensile test technique and ESCA (Electron Spectroscopy for Chemical Analysis) analysis, respectively.

2. Experimental procedure

PAN-based high strength carbon fiber (T300) with a nominal filament dimension of 7 μm was used in this study. The physical and mechanical properties of the fiber from the data sheet of manufacture are listed in Table 1. In order to investigate the influence of thermal treatment on the surface functional groups and mechanical properties, the T300 fiber was thermally treated at temperatures ranging from 600 °C to 1500 °C in a graphitization furnace for 60 min in nitrogen atmosphere.

After thermal treatment, the thermal stability of fibers was characterized by mass loss measurement with an electronic balance (resolution: 0.1 mg). The tensile strength was measured by a single filament tensile technique. X-ray diffraction (XRD) pattern was measured with a Rigaku diffractometer using Cu K α radiation ($\lambda = 0.15418$ nm). From the XRD patterns, the inter-planer spacing and the crystallite dimension of (002) diffraction were calculated. The surface functional groups were measured by ESCA, using a spectrometer employing monochromatic and focused Al K α radiation. The obtained data from ESCA measurement was analyzed by curve fitting using a non-linear least-squares curve-fitting program with a Gaussian–Lorentzian product function. The survey spectrum was collected from 0 eV to 1200 eV, and the binding energies were referenced to the C (1s) line at 284.6 eV.

3. Results and discussion

3.1. Thermal stability of carbon fiber

Fig. 1 shows the mass loss of carbon fibers thermally treated at temperatures ranging from 600 °C to 1500 °C under nitrogen atmosphere. It is clear that mass loss continuously increased with increasing the temperature. At 600 °C and 1500 °C, the mass loss is about 3.2% and 7.9% of original mass, respectively.

At temperature below 600 °C, the mass loss is mainly caused by the decomposition of sizing on fiber surface (normally, the sizing starts the decomposition at 450 °C). With the increase of temperature, the removal of N, H and O atoms from the decomposition of some functional groups in the fibers might be responsible for the mass loss [10–13]. This is quite possible, due to a low carbonization temperature (1200–1500 °C) and a very limited treatment time for the PAN-based high strength carbon fiber fabrication [14].

During the heating process, the fiber could expel impurities as volatile products such as methane (CH₄), hydrogen (H₂), hydrogen cyanide (HCN), water (H₂O), CO₂, NH₃ and various gases [12–14]. Meanwhile, the formation of N₂ has been found to start early at 720 °C and more nitrogen was eliminated from the bulk than from the surface during the heating process [13].

The increase of heating temperature would decrease more the N, H and O in carbon fibers, resulting in a gradual mass loss with

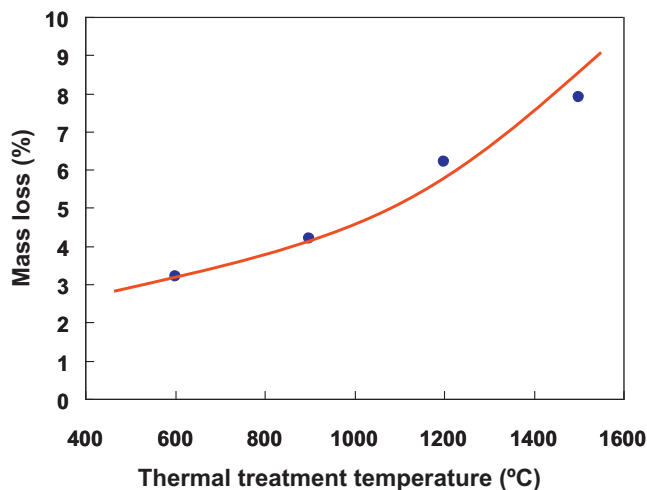


Fig. 1. Mass loss of carbon fibers after thermal treatment at elevated temperatures.

increasing the temperature as shown in Fig. 1. The decomposition of unstable composition and release of gases are not so dependent on the treatment environment [9,14,15]. Furthermore, at high temperatures, the oxidation of carbon fiber due to the presence of impurity such as O₂, CO₂, and H₂O, possibly has a contribution to the total mass loss.

3.2. Microstructure study by XRD

Fig. 2 shows the XRD patterns for the as-received fiber and 1500 °C treated fiber. As a result, the diffraction angles 2θ were around 25° and 43°, which were assigned to (002) and (101) diffraction planes, respectively. The peak (002) is relatively sharp, but the peak (101) is broad.

X-ray diffraction gives an average value of the microstructural parameters by analyzing the entire fiber. It is known that carbon fibers can have an internal structure significantly different from the carbon structure located in the external part [15]. Such structure would result in an asymmetrical regime of diffraction profile. As shown in Fig. 2, a clear asymmetrical peak appears in the (002) diffraction peak at a lower degree position, which indicates that the fiber still has a low degree of graphitization and it must be a complex of turbostratic and graphitic carbons. The turbostratic carbon, which exhibits the same stacked graphene layers with a regular spacing as in graphite but different degree of stacking order [16]. It

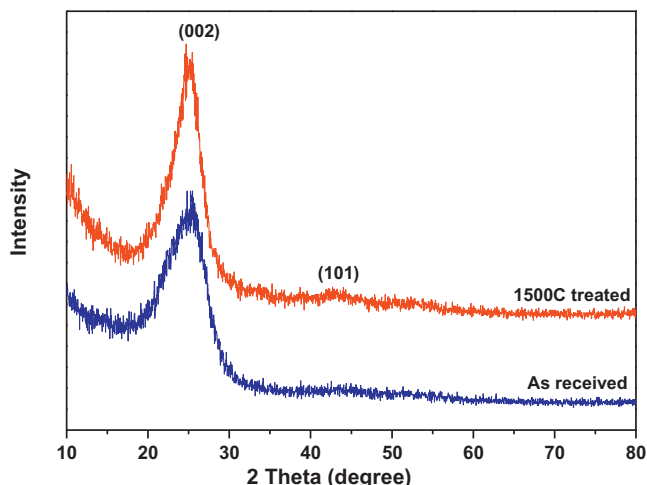


Fig. 2. XRD patterns for carbon fibers after thermal treatment.

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