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Materials Chemistry and Physics 97 (2006) 167–172
MATERIALS CHEMISTRY AND PHYSICS
www.elsevier.com/locate/matchemphys

Effects of ozone method treating carbon fibers on mechanical properties of carbon/carbon composites

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Received 15 May 2005; received in revised form 21 July 2005; accepted 1 August 2005

Abstract

Ozone method was used to modify surface activity of carbon fiber to improve the compressive strength and flexural strength of carbon/carbon composites. The SEM photos of composites fracture shows that ozone method treatment of carbon fiber improved the interfacial adhesion between fibers and matrix, which was the reason of increasing the compressive strength and flexural strength of the carbon/carbon composites. The untreated and treated carbon fibers were investigated by AFM, XPS and the micro-laser Raman spectroscopy, and the contact angles were used to analyze the surface properties of carbon fibers. The results indicated that oxidation of ozone method increased the carbonyl functional group and the surface roughness, changed the graphitization degree of carbon fiber surface and improved the wettability of carbon fibers, so that the compressive strength and flexural strength of the carbon/carbon composites were enhanced. The stronger wettability decreased the formation of traps in the composites, which was one of the major reasons that improved the compressive strength and flexural strength of carbon/carbon composites.
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Keywords: Carbon fiber; Ozone method; Carbon/carbon composites; Properties

1. Introduction

Carbon/carbon composites are often used as structural materials for high temperature applications in electro-mechanical and petrochemical industries because they exhibit excellent corrosion resistance, structural stability and mechanical performance up to very high temperatures, as well as high electrical and thermal conductivity [1,2]. Carbon/carbon composites are carbon fiber reinforced carbon matrix, and it is generally clear that the physical properties of the fibers influence the performance of the composites. Due to their outstanding mechanical properties, carbon fibers are very attractive materials to be employed as reinforcement in composite materials and are applied in all kinds of industries [3–5]. Carbon fibers are included of high modulus (HM) carbon fibers and the high strength (HT) carbon fibers. High modulus carbon fibers are possibly one of the most impressive reinforcements of composites in terms of specific tensile properties. These properties are related to the high degree of orientation of the crystallites and this highly graphitic character is also responsible for high level of thermal and electric conductivity. On the other hand, the enhanced crystallinity of this type of fiber was often reflected in a lower efficiency of industrial methods for increasing the carbon surface activity, in comparison with the high strength (HT) carbon fibers in that HM carbon fibers were more resistant to oxidation than HT ones [6]. In particular, the interface in carbon fiber/carbon matrix composites not only plays the role of transferring the load between fiber and matrix, but also affects the fracture behavior of the composites. To improve the adhesion between fiber and matrix, it is necessary to increase the surface polarity, create more sites for hydrogen bonding and improve the possibility for mechanical interlocking between the fiber materials and the surrounding matrix materials, which lead to good stress transfer from the matrix materials to the fiber ones [7]. In order to improve the fiber–matrix adhesion, many surface treatments were developed which included γ-ray radiation, electrochemically oxidation, plasma treatment, ozone (O_3), etc. [8–11]. In this present study the mechanical

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doi:10.1016/j.matchemphys.2005.08.002

characteristics of carbon/carbon composites and the surface properties of carbon fibers were investigated and AFM, XPS, the micro-laser Raman spectroscopy and the contact angles were used to appraise the surface structural changes of high modulus carbon fibers treated by a ozone method.

2. Experimental

2.1. Ozone method treatment of carbon fibers

Take some carbon fibers and put into ozone calcar. After vacuum handling, the ozone gas was introduced to the reactor at room temperature, and then the reactor was heated to the treatment temperature. After reaction, the samples were cooled to room temperature, and then the reactive gas was purged from the reactor with nitrogen. In the case of the reaction at room temperature, the reactor was cooled and evacuated in a cooling bath prior to charging ozone gas. The reactor was removed from the cooling bath after purging ozone gas with nitrogen. The ozone gas pressure was 0.4 MPa and the nominal reaction time and treatment temperature were adjusted to need. The flow rate of O_3 gas was 0.12 $m^3\,h^{-1}$, and the ozone treatment speed and the distance between valves were 5 mm s^{-1} and 200 mm, respectively. The weight of carbon fibers samples was about 10 g. The ozone method treatment device was shown in Fig. 1.

2.2. Manufacturing of carbon/carbon composites

Composites were prepared by unsized pitch-based carbon fiber using ultrasonic disperse technique for manufacturing prepared mixture with subsequent hot pressing. And, the fabrications were fabricated in a hot-press at 50 MPa at 150 °C for 150 min with a mold method in a conventional composites processing. The fabrications were baked at 1000 °C, and they were baked again after were dipped performs pitch. The fiber volume fraction of bulk specimens was about 7% for all composites.

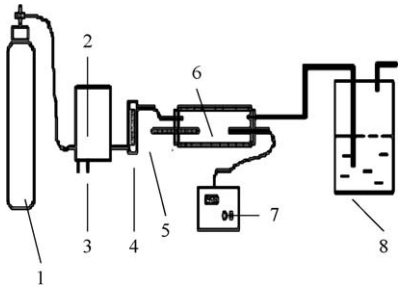

Fig. 1. The structure chart of ozone method treatment apparatus: (1) air tank; (2) generator of ozone; (3) circulation of cold water; (4) flowmeter; (5) thermometer; (6) calcar; (7) thermostat; (8) absorption meter of exhaust gas.

2.3. Analysis method

The effect of the ozone method treatment on the composites fracture morphology was observed using Hitachi S-4700 scanning electron microscopy (SEM). Typical values of voltage and working distance of operation were 2 kV and 8–10 mm, respectively. Carbon fibers were examined on a Russian solver P47 atom force microscopy (AFM). The X-ray photoelectron spectroscopy (XPS), also known as ESCA, measurement of fiber surface was performed with an X-ray photoelectron spectrometer (Perkin-Elmer, PHI 5300) equipped with magnesium X-ray source. The base pressure in the sample chamber was controlled in the range 1028–1029 Torr. The contact angles were measured to get surface energy in order to evaluate the wettability of carbon fiber. The Raman spectra were measured by a JY-T64000 triplegrating spectrometer, using the 514.5 nm line of a SP-165-09 Ar-ion laser with a power of <30 mW, without heat effect. The micro-laser Raman spectroscopy analysis was carried out on the surface areas of interest for the carbon fiber, more attentions were paid to the fiber before and after ozone method treatment.

3. Results and discussion

3.1. Effects of ozone treatment on the compressive strength and flexural strength of the carbon/carbon composites

Carbon/carbon composites were prepared with the untreated and ozone treated carbon fibers. The test results of the flexural strength and compressive strength of carbon/carbon composites, as shown in Fig. 2, indicated that ozone treatment increased compressive strength and flexural strength of carbon/carbon composites. The reason for this would be that the chemical interaction between the carbon fibers and pitch in the matrix and increasing of surface roughness of carbon fibers conduced to the interfaces of carbon fibers and the carbon matrix strengthened. It had been shown

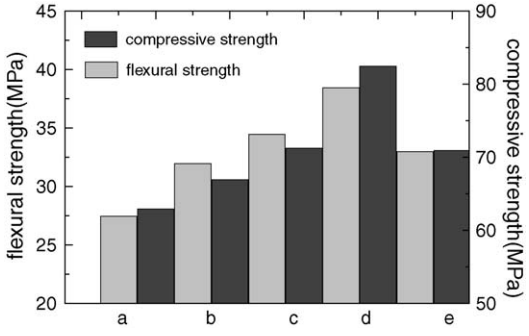

Fig. 2. The compressive strength and flexural strength of the carbon/carbon composites: (a) untreated carbon fiber; (b) the 100 °C for 6 min ozone method; (c) the 120 °C for 3 min ozone method; (d) the 120 °C for 6 min ozone method; (e) the 160 °C for 6 min ozone method.

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