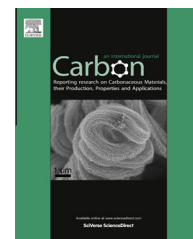


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In situ observations of fractures in short carbon fiber/epoxy composites



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ABSTRACT

In contrast with traditional methods of observation, synchrotron radiation X-ray computed tomography (SR-CT) is an advanced technique that allows direct three-dimensional (3D) and non-destructive observation of microstructures in materials. High-resolution in situ observations (0.7 μm/pixel) of fractures in short carbon fiber/epoxy composites are achieved using the SR-CT technique, and the mechanical load response of short carbon fibers treated with oxidation and those untreated are compared. By the quantitative extraction and analysis of microstructure parameters in high-resolution 3D images, the failure mechanisms of the two materials were studied. The proportion of broken fibers to other types of fiber damage in the sample with oxidation-treated fibers increases by about 6%. Also, the oxidation treatment is able to reduce the ineffective length of the fibers by about 20%, thereby improving the mechanical properties of these composites. The results show that computed tomography can promote characterization of the internal microstructures in carbon fiber-reinforced polymer composites, which will facilitate further theoretical research on the failure mechanisms of these composites.

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1. Introduction

Carbon fiber-reinforced polymer (CFRP) composites have excellent performance characteristics such as a high specific strength, high specific stiffness and good corrosion resistance. The specific strength (i.e., the strength-to-weight ratio) of carbon fiber/epoxy composites can reach 1500 MPa/(g/cm³), which is 10 times greater than normal steel. Therefore, these composites are increasingly used in industrial applications ranging from aeronautics and astronautics to shipping and building, and are especially found in high-technology fields. In researches [1–4] devoted to further improving the performance of these composite materials, treating the carbon fibers by oxidizing them in hot air is one of the commonly used treatments owing to its ease of operation, low cost and

lack of pollution. Hui Zhang et al. [5] found that the flexural modulus and flexural strength of carbon fiber/epoxy composites could be increased by 10% and 21%, respectively, when the carbon fibers were treated with a gaseous oxidation. Lee et al. [6] treated carbon fiber surfaces with a thermal oxidation process in a gas mixture of O₂/(O₂ + N₂), and showed that hydroxyl and carbonyl groups on the surface of these treated carbon fibers led to better interfacial bond strength, thereby improving the composite properties. Ibarra and Panos [7,8] studied the dynamic properties of a butadiene–styrene elastomer matrix composite reinforced with short carbon fibers (SCFs). They concluded that oxidative treatment increased the surface functionality of carbon fibers and promoted bonding with the matrix, leading to further improvements of the composite properties.

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To understand the influence of these improvements on the mechanical performance, it is necessary to study the failure mechanisms of the composites. In previous work, strain and damage evolution in composite materials have been characterized using optical imaging [9], scanning electron microscopy [10] and acoustic emission [11,12], showing that the composite damage usually starts from internal defects. However, none of these methods can produce three-dimensional (3D) and in situ observations of composite material damage. Synchrotron radiation X-ray computed tomography (SR-CT) is an advanced technique that allows direct 3D and non-destructive observation of microstructures in materials [13]. Raz-Ben Aroush et al. [14] achieved direct observation of the fracture process in a quartz-epoxy composite using synchrotron X-ray tomography with an image resolution of $2\text{ }\mu\text{m}/\text{pixel}$. Moffat et al. [15,16] measured crack openings and shear displacements for 0° ply splits in $[90/0]_s$ carbon fiber/epoxy laminates using a computed laminography technique, obtaining 3D images with an isotropic voxel size of $1.4\text{ }\mu\text{m}^3$. Scott et al. [17] reported that the appearance of large clusters in carbon-epoxy laminates occurs within a narrow stress range, instead of accumulating with increasing load. Increased image clarity is important to reveal the fracture process of fiber/matrix composites because it improves the accuracy of composite parameters extracted from the images. For SR-CT experiments, however, there are difficulties in obtaining higher resolution images because increasing the image resolution means decreasing the charge-coupled device (CCD) camera field of view. This creates higher demands on imaging technology, requires the preparation of small samples and increases the complexity of applying micro forces to these small samples at the synchrotron radiation station.

To address these problems, a special tensile testing device is developed in this study that can be used to apply a micro force to small samples at the synchrotron radiation station. The displacement accuracy of this testing device is about $1\text{ }\mu\text{m}$, and the force measurement accuracy is about 0.1 mN . Using this device, high-resolution ($0.7\text{ }\mu\text{m}/\text{pixel}$) in situ observations are obtained showing the fracture process for randomly oriented short carbon fiber/epoxy (SCP/EP) composites. Based on this experimental method, the mechanical load response of two different samples containing untreated SCFs and SCFs treated with oxidation are compared. By using statistics of the distribution of fiber volume fractions in cross-sectional slices along the thickness of the sample, sample areas with low fiber volume fractions seem more likely to break under a load. The proportion of broken fibers to other types of fiber damage in the sample with oxidation-treated fibers increases by about 6%. It is also found that oxidation treatment reduces the ineffective lengths of the composite fibers by about 20%, thereby improving the mechanical properties of the composites.

2. Experiment

2.1. Preparation of samples

Epoxy resin CYD-128, which is a low viscosity ($\leq 2.5\text{ Pa s}$ at 40°C) diglycidyl ether of biphenol-A type resin, and a

hardener (2-ethyl-4-methyl imidazole, EMMZ) used as the matrix were supplied by the Sinopec Group. The composite reinforcements were polyacrylonitrile-based SCFs (supplied by Shanghai Carbon Factory) with an average diameter of $7\text{ }\mu\text{m}$ and an average length/diameter ratio of about 20. For the oxidation treatment, carbon fibers were oxidized in a furnace at 550°C for 1 h, with the condition of constantly pumping air into the furnace [5].

The SCFs and definite amount of curing agent were dispersed in the matrix using a motorized stirrer with stirring speed of 1000 rpm for 30 min at 60°C under atmospheric condition. The mixture was subsequently degassed for 1 h in a vacuum oven at 60°C , and then cast into a stainless steel mold and cured under atmospheric pressure at 120°C for 4 h. Then the samples were cooled to room temperature with natural cooling condition. Two kinds of composites for the observation experiments were prepared: Sample 1 had a tensile strength of 97 MPa and contained a 15% fiber volume fraction of untreated SCFs, while Sample 2 had a tensile strength of 107 MPa and contained a 15% fiber volume fraction of oxidation-treated SCFs. The fibers in all samples are randomly oriented, and five samples were prepared for each kind of composites.

For SR-CT experiments, the image resolution was improved along with a reduction of the CCD camera field of view, which, in this experiment, was approximately $1 \times 1\text{ mm}^2$. Therefore, the sample size was necessarily made correspondingly small. The dimensions of Sample 1 were approximately $0.39 \times 0.36 \times 10\text{ mm}^3$, and the dimensions of Sample 2 were approximately $0.56 \times 0.34 \times 10\text{ mm}^3$.

2.2. SR-CT experiment

Both the carbon fibers and the epoxy resin are weak X-ray absorption materials and have similar X-ray absorption coefficients, making it difficult to get clear reconstruction images of SCF/EP samples with conventional absorption imaging methods. Therefore, phase-contrast (PhC) imaging was used in this work. In general, phase information can be accessed if the X-ray source has a high spatial coherence, which is true for sources in synchrotron radiation facilities like the SSRF in Shanghai. The operation of PhC radiography is essentially the same as traditional radiography with the only significant difference being the necessity of a specific CCD camera-sample distance for PhC radiography [18,19], which is determined by the size of the feature to be identified and the wavelength of the X-rays.

The SR-CT experiment was performed on the BL13W1 beamline in the Shanghai synchrotron radiation facility. The samples were placed at a distance of 14 cm from the CCD camera (14 bit dynamic, 2048×2048 pixel array), which yielded a pixel size of $0.7\text{ }\mu\text{m}$. Seven hundred and twenty radiographs were taken at regular increments over 180° of rotation, each with an exposure time of 1 s and a beam energy of 15 keV.

To apply micro forces to small samples, a special tensile testing device was designed with a displacement accuracy of $\sim 1\text{ }\mu\text{m}$ and a force measurement accuracy of $\sim 0.1\text{ mN}$. Because the tensile testing device blocks X-ray transmission during the rotation in an interval of about 50° , the actual number of usable images is about 500. Therefore, a

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