MEASUREMENT OF THERMAL STRAIN INHOMOGENEITY IN HYBRID CFRP BY IN-SITU FE-SEM

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Abstract (Times New Roman 12 pt, bold, single-line spacing, left-aligned text)

Carbon fiber-reinforced polymer matrix composites (CFRP) is in general having the residual stress and/or strain arise from the differential coefficient of thermal expansion (CTE) of the fiber and the matrix, and the thermal strain also arise from the difference between the longitudinal and the transverse CTE of carbon fiber. Experimental investigation has been made on the effect of anisotropic microstructure of carbon fiber on inhomogeneous thermal strain behavior in CFRP during thermal loading. Nano scale deformation during temperature range of 200K using Joule-Thomson refrigerator stage was investigated by in-situ Field Emission Scanning Electron Microscope (FE-SEM) observation. The measurement of nano scale deformation and strain distribution around the fiber/matrix interface was analyzed by digital image correlation method (DIC) and the transverse and longitudinal CTE of carbon fiber was estimated by analyzed strain distribution.

1 Introduction

Carbon fibers exhibit excellent engineering properties as a reinforcement in composite materials for the aerospace, automotive, infrastructure and sporting applications [1–2]. Carbon fiber-reinforced polymer matrix composites (CFRP) are candidate for space structures due to their high specific stiffness and low coefficient of thermal expansion (CTE). CFRPs are typically fabricated by stacking sequence with multi-plies of different fiber directions, and curing at elevated temperatures under pressure and/or in vacuum. When a CFRP is cooled down to room temperature from the fabrication temperature, the residual stress and/or strain arise from the differential CTE of the fiber and the matrix, and the laminate residual stress also arise from the difference between the ply CTEs in the longitudinal and the transverse directions due to the mismatch in the thermomechanical properties of the fiber and the matrix. Carbon fibers are fabricated from three organic precursor materials of polyacylonitrile (PAN), pitch and rayon followed by a heat treatment. PAN-based carbon fibers generally have high strength, high modulus and low density, and pitch-based carbon fibers tend to have high modulus [3], high thermal and electrical properties with highly anisotropic microstructure [4]. The anisotropic microstructure plays an important role in determining the thermal expansion of CFRP and the delamination at the fiber/matrix interface or the laminate interface during temperature. The measurement method of the thermal expansion of CFRPs and carbon fibers has been shown in the literatures using several experimental techniques [5-8]. However, local strain mismatch at the fiber/matrix interface have not been investigated during temperature.

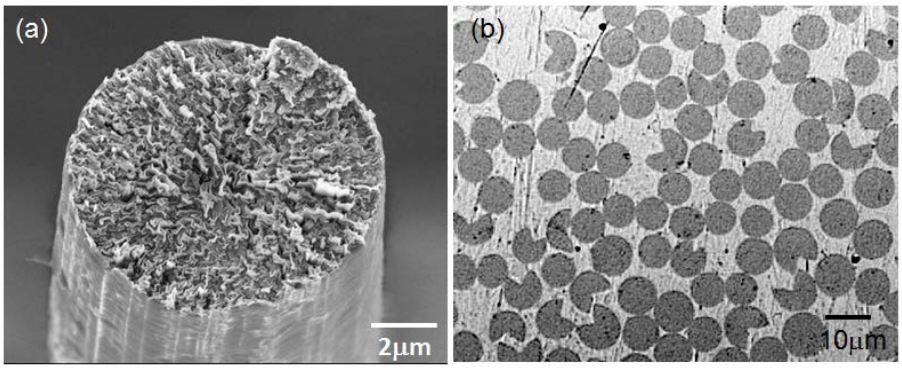

Figure 1. Microstructure of carbon fiber and CFRP: (a) fracture surface observation of K13D pitch-based carbon fiber, (b) carbon fiber reinforced epoxy matrix composite.

In the present study, we have focused on measurement method of nano scale deformation and strain distribution around the fiber/matrix interface in CFRP during temperature via in situ field emission scanning electron microscope (FE-SEM) observations.

2 Experimental procedure

2.1 Material

Carbon fiber used in this study was an ultrahigh modulus pitch-based (K13D) carbon fiber. The K13D pitch-based carbon fiber was supplied from Mitsubishi Chemical Functional Products, Inc. The as-received fibers had been subjected to commercial surface treatments and sizing (epoxy compatible sizing). Figure 1 (a) shows a typical example of the transverse section microstructure of the K13D carbon fiber after observing the tensile fracture surface [3] and the transverse section of the CFRP manufactured by conventional prepreg technology. Inhomogeneous alignment of the carbon fibers can be seen in the figure 1 (b). The CFRP is used as a component for the hybrid CFRP material [9].

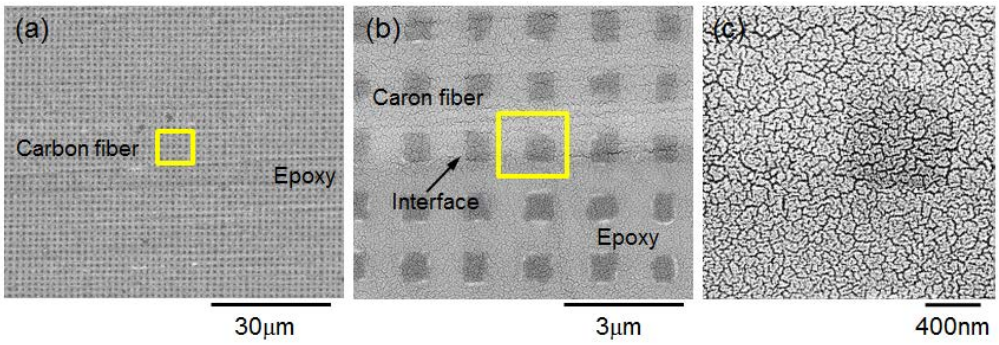

Figure 2. Typical example of a random pattern coted on the sample surface: (a) macro scale observation, (b) near the interface at the magnification of the boxed region in (a), and (c) a nano scale pattern at the magnification of the boxed region in (b).

2.1 Local deformation measurement

In order to measure the local deformation at the fiber/matrix interface, a small rectangular block sample with 1 mm thick parallel to the fiber axis was cut from a unidirectional CFRP. To allow direct observation, one side of the sample was polished with diamond paste up to 0.25 μm. The method on local deformation in materials during

temperature can be measured using several experimental techniques, such as various full-field non-contact optical methods including interferometric, scanning electron microscope grating and digital image correlation (DIC) methods. The DIC method has been widely accepted and used as a powerful and flexible tool for the surface deformation measurement in the field of experimental solid mechanics. The DIC method also has some advantages, compare with the interferometric optical method, such as simple experimental set up and specimen preparation and wide range of spatial resolution. However, the object specimen surface must have a random pattern with high quality image to realize nano-scale deformation measurement. The spattering technique was used to introduce nano-random pattern onto the sample surface [10]. Figure 2 shows a typical example of nano random pattern on the sample observed by FE-SEM with back scatter electron imaging (BSE) mode. The random dots size ranging from 30 to 100 nm are clearly observed and distributed in the whole area. The fiber/matrix interface is also distinguished by BSE mode due to reflectance of backscattered electron.

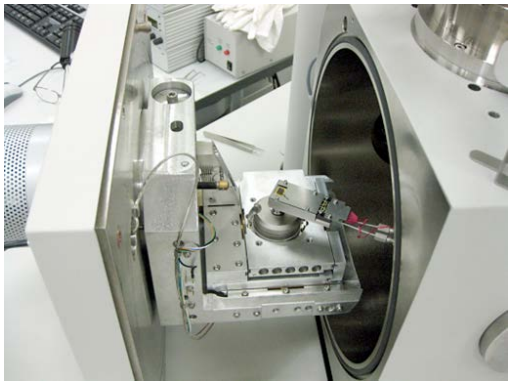

Figure 3. Experimental setup for in-situ FE-SEM installed the heating/cooling stage into the chamber.

A heating/cooling stage of Joule-Thomson refrigerator using nitrogen [11] gas was installed into the FE-SEM (Quanta 200 FEG, FEI Corp.) chamber, as shown in figure 3. In order to measure nanoscale deformation and strain inhomogeneity, digital images of 1024 × 884 with 8-bit value intensity were obtained at various step of temperature by using an in-situ FE-SEM. The temperature of the sample was given at a rate of 10K/min. The sample was cooled down to the initial temperature value of 170K and images were taken at 170, 210, 250, 290, 330 and 370K with a hold time 20 min to stabilize the thermal expansion at each temperature. The images were analyzed using the initial and after thermal loading. The two-dimensional digital image correlation obtained using the commercial software VIC-2D was used to analyze localized displacement and strain distribution at different temperatures.

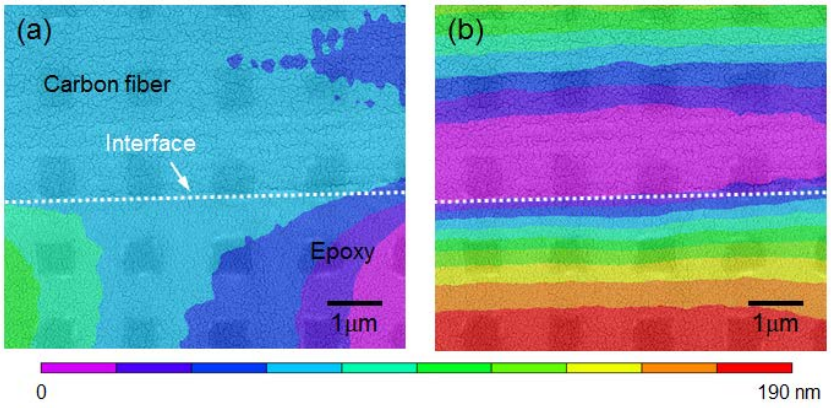

Figure 4. Deformation contour maps in the transverse and longitudinal to the fiber direction: (a) longitudinal and (b) transverse deformation behaviors overlaying BSE image.

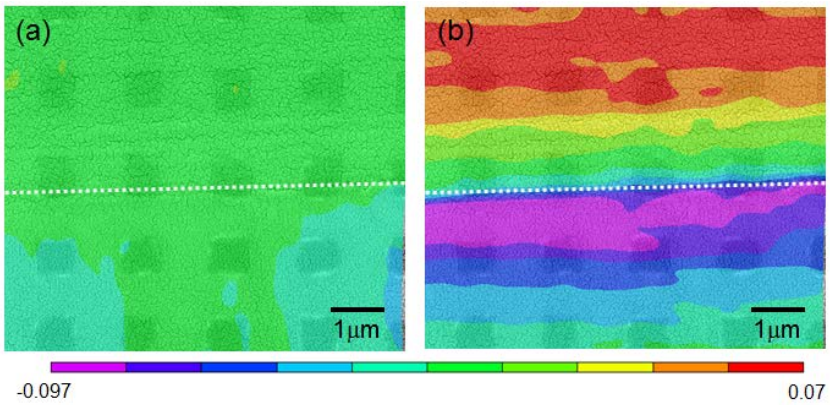

Figure 5. Thermal strain contour maps in the transverse and longitudinal to the fiber direction: (a) longitudinal and (b) transverse strain behaviors overlaying BSE image.

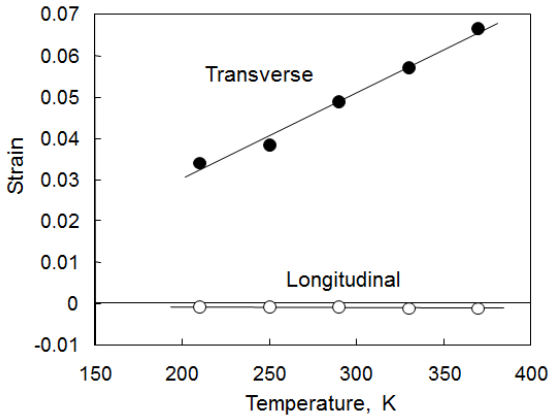

Figure 6. Averaged thermal strain in the carbon fiber versus temperature for transvers and longitudinal directions.

3 Results and discussions

Local deformation at nanometer scale around the fiber/matrix interface in the direction of longitudinal, x, and transverse, y, could be measured by the DIC method. Figure 4 shows an example of the deformation contour maps around the interface at thermal range of 200K in the x and y overlying with BSE image for a selected area of 7.1 × 6.1 μm. The transverse direction of the figure shows fiber direction and the white dotted line indicates the fiber/matrix interface. It is clear that the relative deformation value in the x (103 nm) is approximately 2 times larger than that at the value of y (191 nm). Inhomogeneous deformation behavior is formed in the carbon fiber and near the interface in the epoxy region. This anisotropic and inhomogeneous deformation behavior is caused by the differences of the coefficient of thermal expansion in the transverse and longitudinal directions due to texture microstructure of pitch-based carbon fiber [7]. Figure 5 shows the strain contour map in the direction of x and y. The thermal strain of the carbon fiber in the direction of x is slightly decreased with increasing temperature while the strain distribution in the direction of y is inhomogeneously increased with the temperature. This inhomogeneous strain in the direction of y occurs in the early stage of temperature and is increased linearly with increasing temperature. The compressive strain of approximately 0.08 is locally formed in the epoxy matrix near the interface despite the positive CTE of the epoxy. The transverse and longitudinal strains in the carbon fiber versus loading temperature are shown in figure 6. Each

value is averaged in the fiber direction at each temperature. In the operating temperature, the transverse strain is linearly increased, while the longitudinal strain is slightly decreased with increasing temperature. The slope of strain versus temperature is considered to be an approximate estimate of CTE of the carbon fiber. The transvers and longitudinal CTEs are determined to be 2.1×10^{-4} and -2.35×10^{-6}, respectively. The transverse CTE is much higher than that CTE of pitch-based carbon fiber compared with literature [7]. This suggests that the nano scale deformation and strain distribution in the small area will affect the CTE due to the anisotropy and inhomogeneous microstructure in the carbon fiber. It is considered the transverse CTE after polishing is different with as-received carbon fiber. The present study can be used to provide higher-order of thermal expansion behaviors, such as thermal expansion inhomogeneity and anisotropy, interface delamination, deformation gradients needed for developing the thermal damage mechanism understanding of the CFRP.

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