PIEZO-RESISTIVITY OF TEXTILE COMPOSITES CONTAINING CARBON NANOTUBES AT LARGE DEFORMATION REGIME

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Abstract

Carbon nanotubes (CNTs) have been suggested as a simple and reliable sensor to detect the micro-cracks of polymer matrix composites. Micro-damages of textile composites have been characterized using these CNT networks without visualizing their internal state, concluding that the sensitive observation of micro-cracks is accurate enough to identify the initiation and evolution of internal damages in textile composites. The piezo-resistivity of textile composites is positive at low strain level. However, their piezo-resistivity changes from positive to negative at large deformation regime, even though the micro-cracks of composite are increased. This transition can be observed even in the fiber direction of textile composites. In this study, this phenomenon is examined to determine whether it is physical one or noise, focusing on the microstructure of CNTs network using in-situ X-ray CT.

1. Introduction

Due to their high productivity and mechanical properties, woven textile composites have been used in various industrial fields such as military, civil engineering, aerospace [1]. Recently, the applications of woven textile composites to vehicles or airplane demanding high stiffness and lightweight have been increased due to the development of efficient manufacturing technologies. The reliability of the mechanical properties of textile composites becomes an important issue to facilitate widespread use of textile composite within a target period.

Recently, in-situ monitoring using the piezo-resistive behavior of composite caused by carbon nanotube (CNT) network has been considered as a reliable and nondestructive method that can predict the initiation and evolution of micro-damages precisely [2,3]. In-situ monitoring using CNT networks is based on the electrical conductivity of polymer matrix, which is nonconductive in nature but tailored using CNTs. When a CNT network in matrix or yarns breaks due to micro-damages during deformation, it can signal the damages by changing the electrical conductivity of the matrix. Various attempts have been made to monitor the damage behavior caused by numerous sources such as extension, fatigues or impact, so on. In our previous work, this methodology was applied to 3D braided composites, demonstrating that their piezo-resistive behavior was sensitive enough to detect the damages mechanism [4].

The piezo-resistive behavior refers to a strain-related phenomenon, in particular that the electrical resistivity changes with the strain [5]. The piezo-resistivity itself can be used for measuring strains through the electrical resistance change. Since the damage causes an irreversible change in the material structure, the strain behavior and damage behavior can be measured during the strain sensing by the piezo-resistivity. The piezo-resistive behavior can be quantified by gauge factor as follows.

$$\text{Gauge Factor} = \frac{\Delta R / R_0}{\Delta \varepsilon} \tag{1}$$

In this research, the piezo-resistive behavior of woven textile composite was investigated. To investigate the piezo-resistive behavior of textile composites in various conditions, the fiber orientation and loading condition were controlled during experiment. Ultra high molecular polyethylene (UHMWPE)/epoxy woven composites were prepared using CNTs dispersed in the epoxy matrix. Uniaxial and biaxial tensile tests were carried out and the resistance change of the composites was measured throughput the test. In addition, X-ray computed tomography (X-ray CT) was employed to examine the internal changes of the textile composites [6]. A special testing stage for X-ray CT machine is employed to observe the internal structure of the textile composites during the tensile testing.

2. Materials and Experiments

2.1. Preparation of woven composites

Plain woven preforms made of ultrahigh molecular weight polyethylene (UHMWPE) (Dong Yang Rope MFG in Korea, fiber modulus and strength are 111 and 3 GPa, respectively) were used as a reinforcement. The woven preforms were laminated for 4 layered composites with a stacking sequence of $(0/90)_4$ (on-axis specimen), $(\pm 45)_4$ (biased specimen), and (22/85/-85/-22) (off-axis specimen), respectively. Epoxy resin (Epofix, Struers) was used as a matrix. Multi-walled CNTs (MWCNTs, Hanwha Nanotech in Korea, diameter: 15~20 nm, length: 10~15 micron) were dispersed into the epoxy matrix before curing using a three-roll-mill calendaring. The curing agent was then added to the slurry of epoxy and CNTs by additional three-roll-mill calendaring. Vacuum-assisted resin transfer molding (VARTM) was carried out to fabricate the woven composites.

2.2 Tensile testing and characterization of the piezo-resistive behavior

Two kinds of specimens were prepared to characterize the piezo-resistive behavior of woven composite under uniaxial and biaxial tensile loading (Figure 1). For the uniaxial tensile test the dog-bone shaped specimens were prepared. Electrodes were formed at both end of the gauge region using silver paste and conductive epoxy resin (CW-2400, Chemtronics) containing silver particles. The uniaxial tensile test was performed using a tensile tester (INSTRON 8516) in the displacement-control mode. The head speed was set to be 0.021 mm/s. An electrometer (Keithley 6517B) was used to measure the electric current during the test using a synchronized data acquisition system.

For the biaxial tensile testing, cruciform specimens were prepared. Note that four electrodes were formed on the cruciform specimen to measure the electric current in the two directions during the biaxial tensile testing. The biaxial tensile test was then performed in the load-control mode. The load ratio in the x and y direction was set up to 2:1, i.e., F_x:F_y=2:1 throughout the testing. The loading rate was 4 kN/min on the x axis. The electric current in the two directions during the biaxial tensile testing was measured using the same electrometer and data acquisition system as used for the uniaxial tensile test

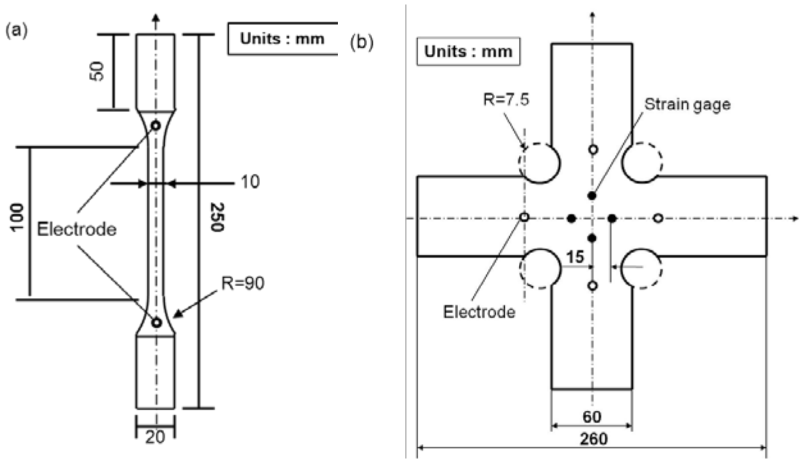

Figure 1. Schematic view of specimens (a) uniaxial tensile testing specimen (b) biaxial tensile testing specimen.

2.3 In-situ observation with tensile test

A special specimen was designed for in-situ tensile test in X-ray CT (Figure 2). A round-shaped notch was introduced at the center of the specimen that can induce the stress concentration and observe the damage behavior at a controlled manner. CT scanning was performed at several strain levels. A micro-CT (Skyscan 1172, Belgium) was used. Scanning conditions were 40 kV and 200 μA. The scanned images were saved in TIFF format and were reconstructed through a analysis program provided by the manufacturer. A specially designed testing stage with 200 N of load cell was used for in-situ tensile testing. Note that for CT scanning the loading was stopped and the specimen was maintained between the grips. This might induce the viscoelastic relaxation due to long scan time (about 40 ~ 60 mins).

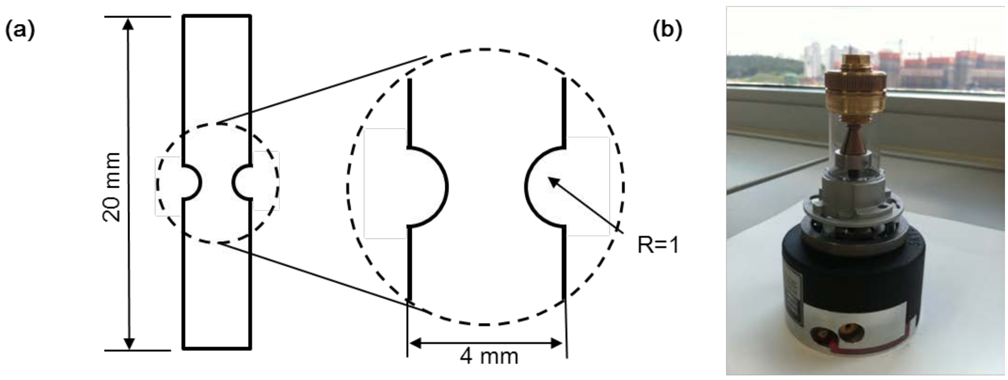

Figure 2. (a) Schematic view of *in-situ* tensile specimen by X-ray CT and (b) a testing stage employed in this study.

3. Results and Discussion

3.1 Tensile testing and characterization of the piezo-resistive behavior

Figure 3 (a) shows a typical mechanical and piezo-resistive behavior of the textile composites. The mechanical behavior of the woven composite can be classified into four stages by considering the slope change of the stress and strain curve. Typically, the stress increases linearly in the stage 1, followed by the slop decrease in the stage 2. The slope increases again in the stage 3, implying that there are strengthening mechanisms working in the woven composite. In the stage 4, the composite finally reached to the fracture.

The electrical resistance was increased over the stage 1 and its slop was decreased through stage 2. The piezo-resistive behavior was nearly linear in the stage 1. The stage 2 can be further divided into four sub-stages according to the slope change of resistance, which are denoted by stage 2-1, 2-2, 2-3, and 2-4 (Figure 3 (b)). Interestingly, the resistance change started to decrease upon the stage 2-4. The reduced resistance change can be explained by the yarn compaction and Poisson's effect. Detailed discussion will be provided at the Conference.

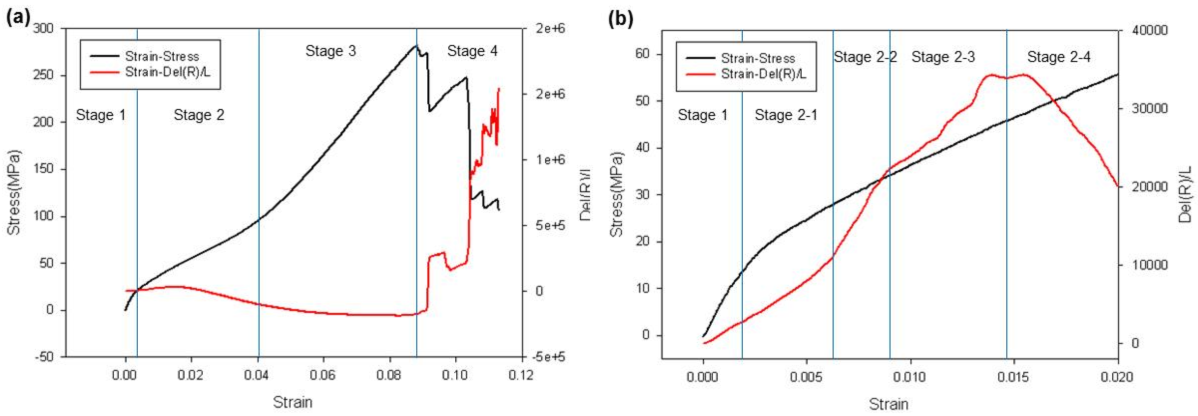

Figure 3. Mechanical and piezo-resistive behavior of on-axis woven textile composite (a) overall scale (b) expanded small strain regime.

3.2 In-situ observation with tensile test

Figure 4 shows micro-CT images of woven textile composite obtained from on-axis tensile test in CT machine. The yarn, epoxy matrix and cracks can be distinguished by different gray-scale. At zero strain, the composite showed some voids, which were introduced probably due to the infusion of air bubble in VARTM process or laser cutting for preparing specimen (Figure 4(a)). The damage state of the composites was remained without significant changes through the elastic region (0.2 % strain, Figure 4 (b)). The damages such as fiber-matrix debonding and transverse crack were observed in the composite at a strain of 1.5 % (Figure 4(c)). At relatively large strain regime (e.g, 2.8 %, Figure 4 (d))), however, damages seemed reduced, which may explain the reduced electrical resistance observed in Figure 3. Detailed explanations and more analysis of this in-situ tests will be presented at the Conference.

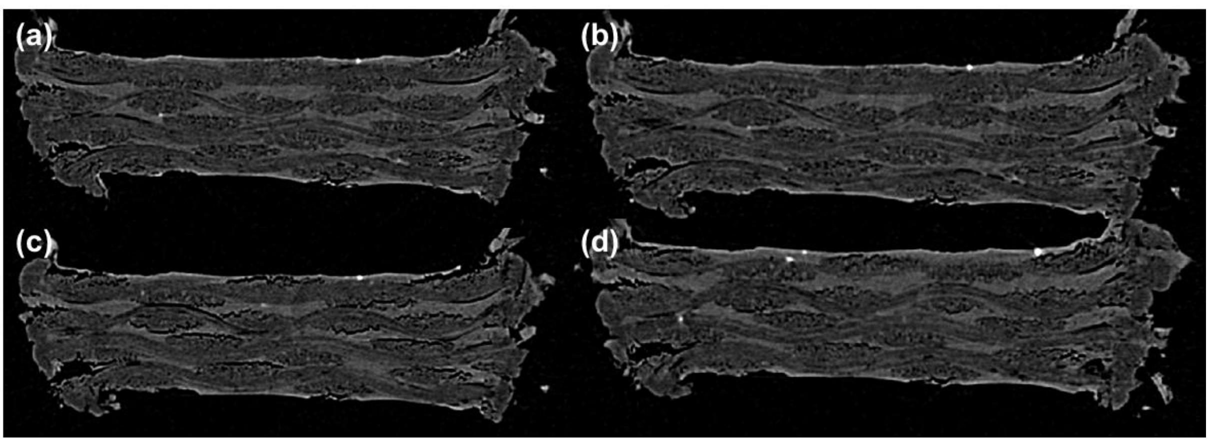

Figure 4. X-ray CT images of on-axis woven textile composite (a) un-stretched strain (reference state) (b) 0.2 % of strain state (c) 1.5 % of strain state (b) 2.8% of strain state

4. Conclusions

The piezo-resistive behavior of woven textile composites under tensile loading was investigated using CNT conductive networks and *in-situ* X-ray CT. The resistance change observed according to the loading directions was sensitive enough to identify damage mechanisms occurring in the woven composites in the small strain regime. After certain strain the negative piezo-resistive behavior was observed by significant Poisson's effect and yarn compaction and rotation. The microstructural changes in the textile composites were investigated by in-situ X-ray CT test. This novel experiment confirmed that the micro-cracks such as transverse crack and fiber-matrix debonding were reduced at large strain regime, explaining the negative piezo-resistive behavior of the woven composites.

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