Self sensing glass/epoxy composites using carbon nanotubes

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SUMMARY

This paper presents results of an investigation on the sensitivity of carbon nanotube network in detecting the presence of cracks in the laminated composites. The results show that the carbon nanotube network is more sensitive than strain gauges in crack detection.

KEYWORDS: Cracks; Fatigue; Carbon nanotubes; Electrical resistance; Sensing

ABSTRACT

There has been recent interest in the investigation of the change in electrical conductivity of carbon nanotube network incorporated in polymeric composites, particularly glass/epoxy. The findings show that electrical conductivity changes significantly corresponding to the load increase in the laminate, and there are sudden changes when the laminates are subject to failure. In this work, crack detection under fatigue loading was investigated using a network of carbon nanotubes within an epoxy-based composite, reinforced with bidirectional glass fibres. Strain gauges have also been used. Three types of samples were prepared: samples without any imposed defects, samples with a cut on the cured sample that is far away from the strain gauge, and samples with a cut in the mid-layer of glass fibre (before curing). The results show that the carbon nanotube network is more sensitive in detecting the occurrence of cracks than strain gauges.

INTRODUCTION

Carbon Nanotubes (CNT) are known as the stiffest and strongest materials in the world [1-3]. They were discovered in the early 1990s and researchers started to focus on these new materials due to their considerable physical and mechanical properties. Their remarkable mechanical properties such as high stiffness and strength, exceptional resilience, low density, fiber-like structure with high aspect ratio (length/diameter), as well as high electrical and thermal

conductivity render Carbon Nanotubes (CNTs) potential as nanoscale reinforcement to achieve improved electrical properties [4-9]. Recently, researchers have utilized them as strain sensors by embedding them in polymer matrix composites (PMCs) and monitoring damage and subsequently failure, by direct measurement of current in the composite [10-13]. Damage monitoring using electrical techniques is of long term interest to researchers and began with the use of carbon fibers as the conductive reinforcement, so that fracture of the fibers would result in a change of electrical resistance [14-16]. Since matrix-dominated fracture mechanisms can not be monitored utilizing these conductive fibers and this also places a restriction on using just conductive fibers such as carbon fibers, using a dispersed conductive reinforcement in the matrix has become the main consideration for damage monitoring. The high aspect ratio of carbon nanotubes has been considered as crucial to the formation of a conductive percolating network through the polymer matrix composite at relatively low CNT concentrations: such a network is highly sensitive to the onset of damage in matrix-dominated fractures [10]. This exceptional sensitivity causes an increase in electrical resistivity along embedded carbon nanotubes in PMCs under even a low mechanical load [17]. In comparison with resistance-type strain gauges, which provide a conventional way for measuring strain, using conductive CNT may not have the same limitations, such as measuring strain at only specific locations and a lack of versatility and flexibility [18]. In Thostenson and Chou's study, [10] unidirectional glass fiber-epoxy composites with dispersed multiwall carbon nanotubes within the epoxy have been utilized to evaluate the damage and percolation threshold in tensile and flexural tests as an in situ sensor. When considering non-static loads such as fatigue, which is one of the most crucial factors for failure in many structural components, the long term durability of polymer matrix composites is highly dependent on the polymer matrix and the fiber-matrix interface [19]. In other words, fatigue life and damage tolerance are strongly affected by matrix cracks, like micro cracks appearing between fiber reinforcements, or ply delamination between layers [10]. Carbon nanotubes as additives can play a significant role as distributed sensors to monitor damage and to determine the extent and defect propagation created by cyclic loads [20, 21]. This aspect is further explored in this study. In another study, Böger et. al. [22] worked on stress/strain and damage monitoring of glass fiber-epoxy using carbon nanotubes and carbon black during tensile, fatigue, and interlaminar shear strength testing. Damage monitoring and detection of failure location is further explored in this study during cyclic loadings.

EXPERIMENTAL

Primary materials

Multiwall carbon nanotubes grown by the chemical vapor deposition technique (more than 95% purity) have been used in this work (Cheap Tubes). They have diameters in the order of 10-20 nm and lengths of 10-30μm. The epoxy polymer and curing agent are bisphenol-F epoxy, EPIKOTE Resin 862, and EPIKURE W (an aromatic amine curing agent), respectively (Hexion specialty chemicals). Bidirectional woven glass fibers have been utilized as the mechanical reinforcement.

Fabrication of nanotube-fiber-epoxy composites

Due to agglomeration of the nanotubes, in order to untangle and disperse them within the epoxy to make the network of sensors, a calendering machine (Three Roll Mill EXAKT) has been used to produce high shear force mixing while passing the resin/nanotubes mixture through the rolls. The minimum agglomeration was seen after gradually reducing the gap setting from 50μm to 5μm. 1wt% of MWCNTs was dispersed within the epoxy resin due to conductivity achievements recorded elsewhere [23]. The resin was then heated up to 50^{o}C to reduce the viscosity and the curing agent (in the ratio of 26.4:100) was added to the epoxy. After stirring for 5 minutes, the mixture was heated up to 70^{o}C for 20 minutes in a vacuum oven in order to degas and remove the bubbles. The final resin obtained was applied to two layers of bidirectional woven glass fibers by hand lay-up followed by two hours of vacuum bagging. The fabricated polymer matrix composite was cured in an oven for 6 hrs at 130^{o}C.

Electrical measurements under fatigue loads

Samples were cut out of the cured composite plate, according to ASTM D 3039 for fatigue testing. To improve gripping of the end-tabs of the samples, screen sandpaper was bonded onto the tabs. Silver epoxy-based glue was used as the contact for the conductive probes for electrical resistance measurement. The measurements were made using a high resistance meter (Agilent machine 4339B). Fatigue tests were carried out on an MTS 100KN universal testing machine and at the same time, a constant source voltage was applied to the samples and the electrical current was measured in order to calculate the electrical resistance change. For fatigue samples, residual resistance change has been measured as $(R_i\text{-}R_0/R_0)$ x 100 where R_0 is the electrical resistance of the measured region before loading, and R_i is the resistance while the sample is unloaded.

RESULTS

Monitoring damage in fatigue testing

The observed sensitivity of the carbon nanotube network in indicating the state of deformation in the composite samples gave rise to the motivation to investigate its ability to monitor damage which has been studied in our previous work by using static loadings [24]. For this study, three electrical probes were bonded to the sample providing two regions of equal dimensions (each region about 3.75 cm long) in the gauge area (Figure 1). Three types of samples were made and tested during dynamic cyclic loadings:

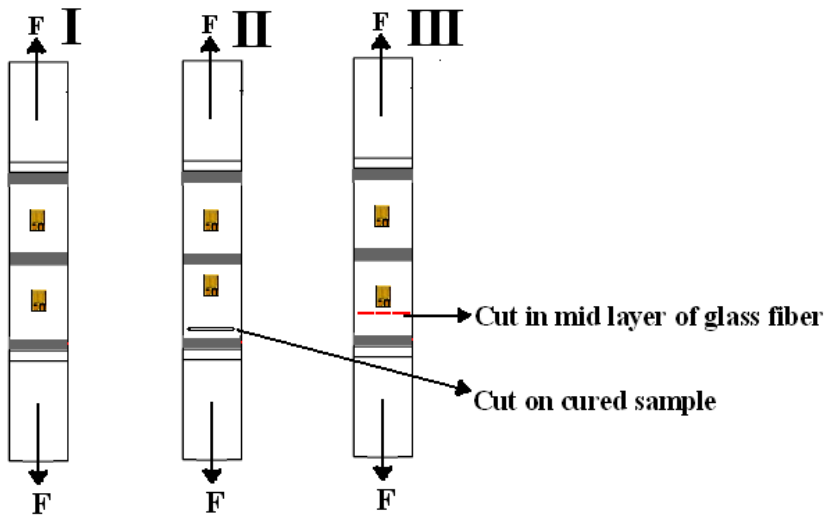

Fig. 1: Three types of samples for damage monitoring in two regions in fatigue test.

Type I: Five samples (two layers of fabrics) with two regions with strain gauges placed in the center of each region.

Type II: Two samples (two layers of fabric) with two regions with a deliberate cut made in the lower region of the cured sample. The first strain gauge is attached at the center of the top region and the other one in the bottom region, nearer the top region to allow some distance from the defect.

Type III: Two samples with three layers of glass fiber rather than two. The mid layer has a cut in the bottom region before curing. After manufacturing of the composite, the defective part of the sample was marked in order to identify the artificial crack made in the mid layer. Strain gauges were positioned at the center of each region. The strain gauge in the lower region is located about 2-3 mm above the crack location.

Maximum cyclic loads of 4500 and 6000 N were applied for type I samples, 2000 and 3000 N for type II samples, and 6000 N for type (III) samples. After every four cycles of loading, the sample was unloaded and electrical resistance and strain values were measured. After a number of cycles, a tensile test was conducted to find the exact failure location.

Fatigue testing of type I samples:

In type I, five samples were tested. Figure 2a shows the responses for sample 3 with a maximum load of 4500 N. After conducting the tensile test done after the fatigue test, failure occurred between points 1-2 which was very close to the upper strain gauge (almost 1-2 mm). In this case both residual strain and residual change in resistance show higher values for the region between points 1-2 (as compared to the region between points 3 and 4), which corresponds to the location of the crack. Figure 2b shows the response for sample 5, with a maximum load of 6000 N. The crack occurs in the region between points 2 and 3. In this case, the strain gauge values in the two regions do not differ, while the residual change in resistance in region 2-3 is much higher than that in region 1-2. This result shows that the residual change in resistance corresponds better with the crack location. The results from the other 3 samples of type I also show similar pattern. Strain gauge values sometimes correspond to the location of the

crack, and sometimes not. On the other hand, residual change in resistance always corresponds to the location of the crack.

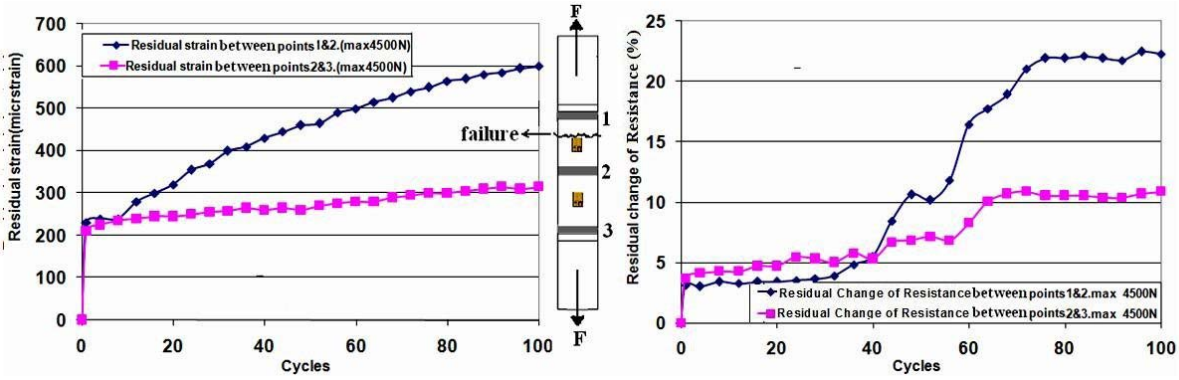

Fig. 2a: Residual change of resistance and residual strain in two regions, after 100 fatigue cycles with maximum load of 4500 N (type I, sample 3)

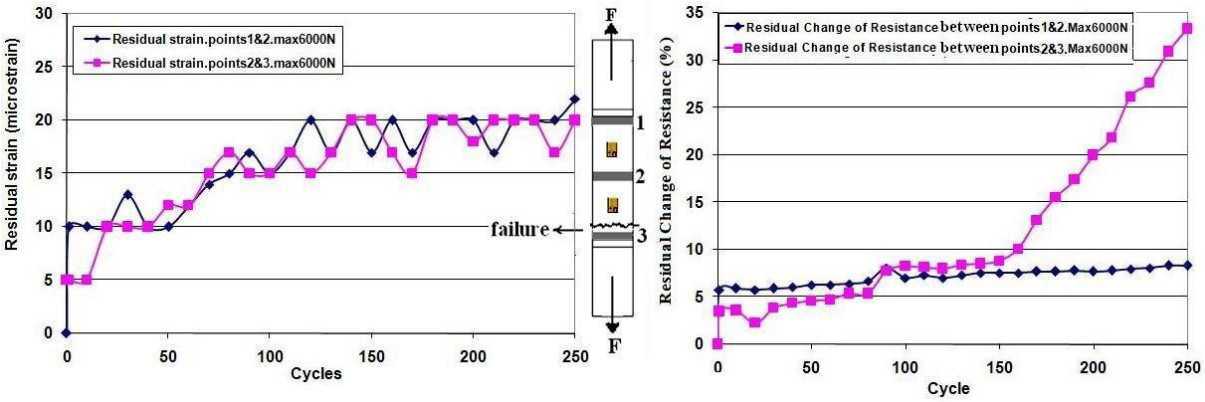

Fig. 2b: Residual change of resistance and residual strain in two regions, after 100 fatigue cycles with maximum load of 6000 N (type I, sample 5)

Fatigue testing of type II samples:

Similar tests were carried out on 2 samples of type II. Since a cut has been made in the lower region between points 2-3, to decrease the effect of local deformation around that cut, a strain gauge was placed at a distance of 2.5 cm from point 3 (Figure 3a and 3b). Fatigue testing was implemented with maximum cyclic loads of 2000 and 3000 N, knowing that the failure would occur in the defective part. As shown in Figures 3a and 3b, the resistance measurements show higher rate of change between points 2 & 3 in which the failure initiation takes place.

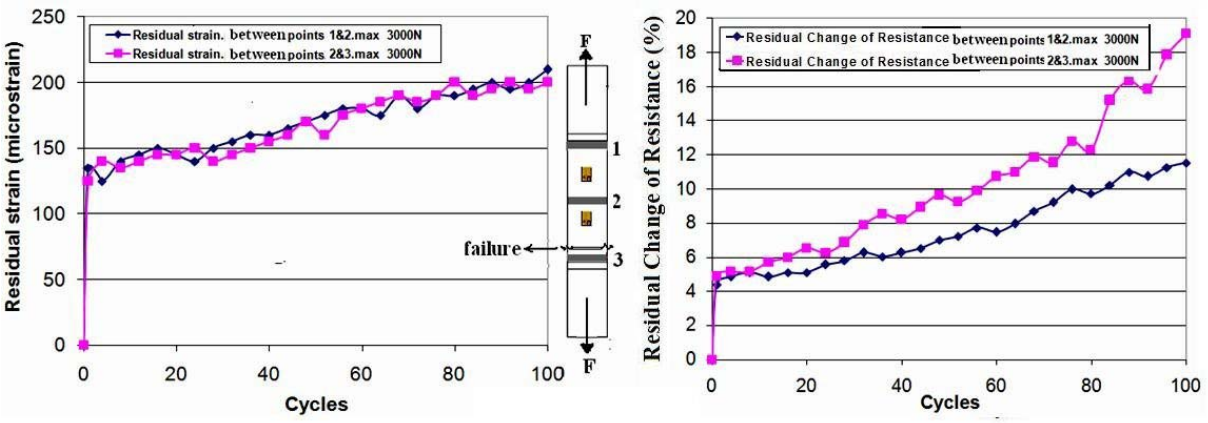

Fig. 3a: Residual change of resistance and residual strain in two regions, after 100 fatigue cycles with maximum load of 2000N (type II, sample 1)

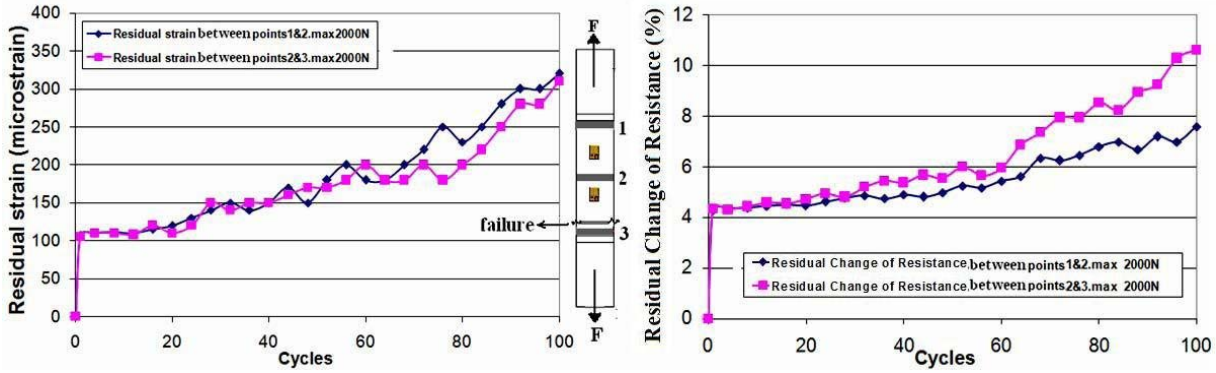

Fig. 3b: Residual change of resistance and residual strain in two regions, after 100 fatigue cycles with maximum load of 3000 N (type II, sample 2)

Fatigue testing of type III samples:

Two samples of type III as depicted in Figures 4a and 4b were fatigued with a maximum load of 6000 N. The residual strains between points 2-3 increase more than that between points 1-2. The resistance increases show the same behavior.

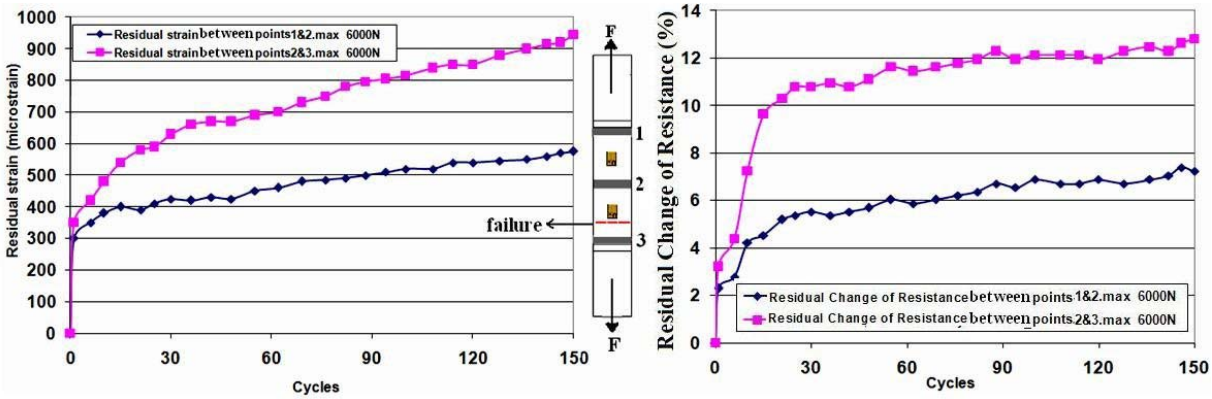

Fig. 4a: Residual change of resistance and residual strain in two regions, after 150 fatigue cycles with maximum load of 6000 N (type III, sample 1)

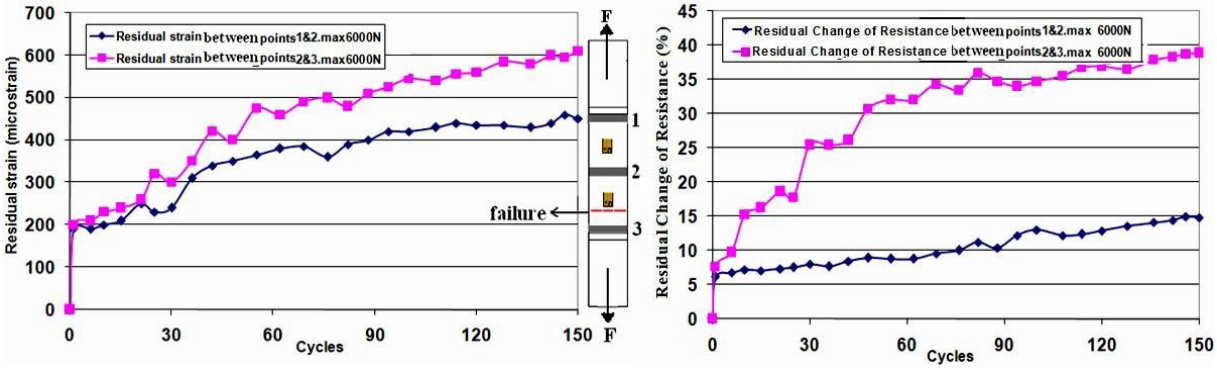

Fig.4b: Residual change of resistance and residual strain in two regions, after 150 fatigue cycles with maximum load of 6000 N (type III, sample 2)

In order to see whether the increase in residual change in resistance corresponds to any damage in the material, some samples that were tested to a maximum of 3500 N after 100 cycles were cut along the longitudinal direction. The sections were polished and observed under microscope. Figure 5 shows two of the sections. It can be seen that matrix cracks occur in these samples.

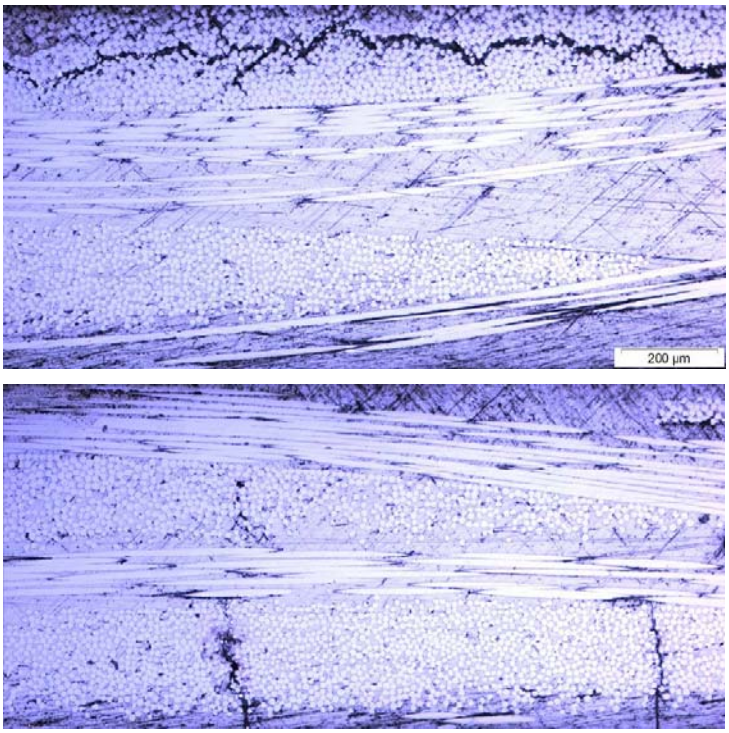

Figure 5: Micrographs of sections of samples that were tested under fatigue loadings (Both samples have same scale)

DISCUSSION

Better sensitivity of the carbon nanotube network in the monitoring of damage detection as compared to strain gauges is illustrated from the experimental results.

The better sensitivity of the carbon nanotube network in detecting damage is supported by more sensitive residual change in resistance coupled with the presence of matrix cracks for loads where there is large change in residual resistance and little change in residual strain [25]. This is further supported by fatigue results done on samples without deliberate cracks (Figures 2a, 2b) and samples with manufactured cracks (Figure 3a, 3b, 4a, 4b). The better sensitivity of the carbon nanotube network as compared to strain gauges can be explained by the fact that carbon nanotubes are spread throughout the matrix in the composites, and most of the initial cracks and delaminations take place within the matrix material. Strain gauges can sometimes reveal the presence of cracks and sometimes not. This depends on the orientation of the cracks as compared to the orientation of the strain gauge. If the crack happens to be delamination of fibers parallel to the strain gauge length direction, then the strain gauge may not pick it up. If the crack orientation is normal to the direction of the strain gauge, then it is more probable that the strain gauge will feel the crack. In addition, whether the strain gauge will feel the presence of the crack depends on the distance from the gauge to the crack and on the ductility of the material. The stress path around a crack may go around the gauge if the strain gauge is located too close to the crack. On the other hand, since the nanotube networks are connected all over the sample, the occurrence of any defect or damage can cause disconfiguration of the nanotube network. This in turn produces an increase in resistance along the sample, regardless of the location of failure.

The above points show that carbon nanotubes have the potential to monitor the strength degradation during dynamic loading and to predict failure before it happens. This self-sensing method using nanotubes as a network of sensors can also be proposed for precise failure location prediction in polymer matrix composites.

CONCLUSION

Towards the development of a new method to monitor failure under dynamic loads, multiwall carbon nanotubes were incorporated in an epoxy matrix to form a network of sensors. By applying dynamic cyclic loading and comparing the resistance change with strain gauge measurements in different zones, it was seen that the electrical resistance measurements show more consistency in correlation with failure location. Failure location can be predicted by higher increase of resistance change in a region compared to other regions of the sample. The formation of matrix cracks causes disconfiguration of the nanotube network and subsequent reduction of the electrical current flow through them. These results show that nanotube networks have the potential to be used for monitoring the integrity of composite laminates during both static and cyclic loads.

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