Piezoresistive Behavior of Epoxy Matrix-Carbon Fiber Composites with Different Reinforcement Arrangements

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ABSTRACT: In this work, the self-monitoring capability of epoxy matrix-carbon fiber composites has been studied. Different concentrations and arrangements of reinforcements were used, including random chopped, unidirectional and bi-directional continuous carbon fibers, weaved and nonweaved. Mechanical properties were determined by uniaxial tensile tests. The composite electric to mechanical behavior was established by determining its electrical resistivity variation as a function of the stress-strain curve. It was observed that the composites electrical resistance increased during tensile tests, a trend that indicates piezoresistive behavior. The increase was linear for the chopped reinforced composites, while it exhibits different slopes in the continuous reinforced composites. The initial smaller slope corresponds mainly to separation of the 90° oriented fibers and/or transversal cracking of the matrix, whereas the latter higher slope is caused by fiber fracture. The results demonstrated how each reinforcement configuration exhibited a unique and typical electrical response depending on the specific reinforcement, which might be appropriate either for strain-monitoring or damage-monitoring. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 111: 2851–2858, 2009

Key words: composites; reinforcement; structure-property relations; mechanical properties; strain

INTRODUCTION

Self-monitoring materials may be considered smart materials because they are capable of sensing their own deformation and damage. In fact, self-monitoring structural materials are intrinsically smart, because there is no need to attach or embed any sensors to the structure. The advantages of these materials compared with those that require external sensors are as follows: lower cost, greater durability, large volume monitoring capability, and lack of detriment in mechanical properties.[1]

The self-monitoring capability of the polymer-matrix composites reinforced with carbon fibers is based on the dependence of electrical resistance on strain and fracture. To have a composite with this ability, one of the components, usually the fiber, should be an electrical conductor, while the matrix should not be highly conductive, otherwise the high resultant conductivity would result in electrical resistance changes too small to be detected. In general, these composites possess electrical conductive fibers which besides increasing the electrical conductivity of the material, also improve its mechanical resistance.[1–3]

Several studies conducted on single carbon fiber electromechanical response indicate that the electrical resistivity increases with tensile strain and decreases with compressive strain, in a reversible manner. This effect is mainly due to dimensional changes experienced by the material and is apparently not due to resistivity changes. This behavior is observed in the elastic regime of the material, where the deformation is completely recovered upon unloading. However, fiber damage may occur prior to fracture causing an irreversible deformation, which corresponds to the plastic regime. In this case, the damage in the fibers will increase irreversible the electrical resistivity of the material.[4,5]

In the case of unidirectional fiber reinforced laminates submitted to tensile loading, a reversible decrease in resistivity has been observed during the early stages of the deformation process.[1,6,7] This is attributed to an increase in fiber orientation in the

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stress direction. Upon further loading an irreversible electrical resistivity increase is observed, mainly caused by fiber damage, which produces a progressive reduction of electrical pathways throughout the material. In chopped carbon fiber reinforced polymers, the applied tensile load produces an increase in the separation between neighboring conductive fibers with a consequent increase in the overall electrical resistivity. On the other hand, under compressive loads, the distance between fibers is reduced, and therefore, a decrease in electrical resistivity is observed.

It has been previously shown that carbon fiber reinforced epoxy materials could be used in structures that would be able to sense strain in real time, because of their piezoresistive capability.[4,5,8] However, every composite type has been evaluated independently, and to the authors' knowledge a comprehensive study comparing the piezoresistive response of different reinforcement arrangements has not been presented. The knowledge of how different carbon fiber reinforcements perform as self-monitoring structures is vital for widely different applications. For instance, continuous carbon fiber reinforced materials are suitable for infrastructures, like offshore platforms, tanks, and pipelines; whereas chopped carbon fiber reinforced materials could be successfully used as self-monitors in coatings applied to surfaces of already operating structures. Moreover, the advantage of a comparative study of the different types of carbon fiber reinforcements is to establish the relationship between radical changes in electrical resistance and matrix cracking, which occurs prior to the overall composite failure.

The aim of this work is to present a comparative study of the piezoresistive behavior for composites of different content, configuration, and orientation of reinforcement, to acquire fundamental knowledge to allow the prediction of the piezoresistive response for any specific laminated composite. The results provide the bases for designing the optimum fiber arrangement that allows adequate monitoring capability according to specific applications, based on both mechanical properties and self-monitoring capability.

MATERIALS AND METHODS

Continuous reinforced composite samples were constructed from carbon fiber unidirectional or bi-directional fabric manufactured by Zoltek (St. Louis, MO). Carbon fiber products PANEX® 33 UD21 and PANEX® 33 PW28 (both made from continuous carbon fiber PANEX® 33 48K), were used to fabricate the composite panels. Table I shows typical properties of these carbon fiber fabrics. Chopped carbon fiber reinforced composite samples were manufactured with short fibers (6 mm, resistivity 1.72×10^{-5} Ω-m, PAN based) provided by Nichimen Paltex Corp. (Japan). The material used as matrix was epoxy resin (RESIPOX 5928 from Resimon) cured with a cicloaliphatic amine (EPICURE 3370 from Shell Chemical Co.) according to manufacturer recommendations. All laminates have two fiberglass mat layer (type C) on each surface, used to improve the surface quality of the chopped carbon fiber reinforced laminates. The same configuration was used in the continuous reinforced composites for the sake of comparison.

TABLE I
Properties of the Employed Carbon Fiber Reinforcements

Property	PANEX® 33 UD21	PANEX® 33 PW28
Tensile strength	3,800 MPa	3,800 MPa
Tensile modulus	228 GPa	228 GPa
Electrical resistivity	0.00155 Ω-m	0.00155 Ω-m
Fiber diameter	7.2 μm	7.2 μm
Carbon content	95%	95%
Type of fiber arrangement	Unidirectional	Plain weave
Weight	722 ± 10 g/m^2	949 ± 10 g/m^2
Thickness	0.102 cm ± 10%	0.165 cm ± 10%
Filament content per tow	45,700	45,700

Continuous carbon fiber reinforced composite laminates were laid up in a 15 × 25 cm^2 flat compression mold. The layers were stacked in the mold with the uncured resin mixture and a mold release spray was applied to the mold surfaces. The laminates were cured for 24 h at room temperature and under 32.7 kPa compressive pressure. Chopped carbon fiber reinforced composites were fabricated by mixing the fibers with the epoxy resin manually with a glass rod, putting the mixture in the mold, and then cured as described before. A scheme showing the different composites fabricated is presented in Figure 1.

The volume fraction of carbon fibers in each composite is shown in Table II. For specimen preparation, glass fiber reinforced epoxy end tabs were applied to both ends on each side of the laminates. The laminates were cut into tensile test samples, with the dimensions specified in ASTM D-3039, with a constant thickness of 3 mm for all laminates.

Electrical contacts were accomplished in two different ways. For continuous fiber reinforced composites, a low-loss wire (copper coated with nickel) was attached to each sample end, covering the entire cross section, with silver paint coating. For chopped fiber reinforced composites, the low-loss wire was attached to a stainless steel screw (diameter: 3 mm, length: 50 mm), which was screwed in a perforation made through the cross section of the sample

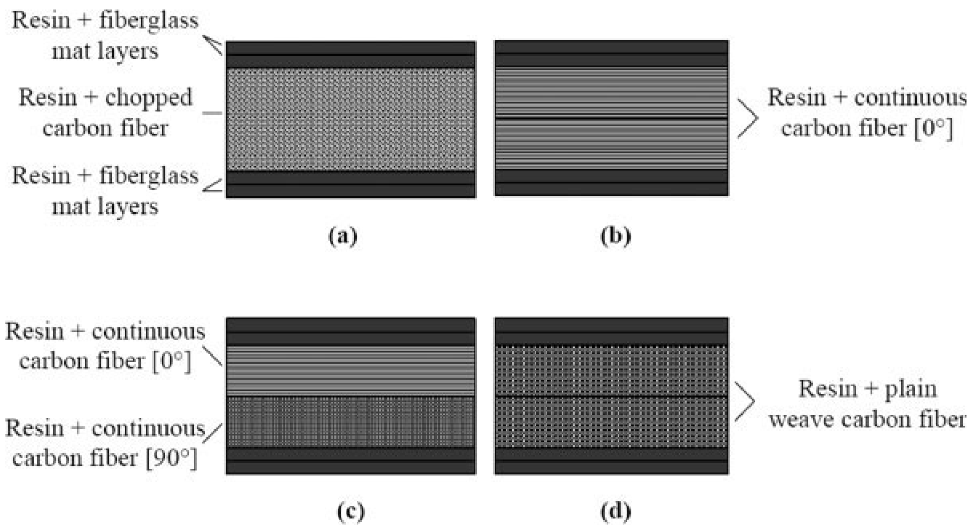

Figure 1 Scheme of the composites fabricated: (a) chopped carbon fiber composite, (b) continuous unidirectional carbon fiber composite, (c) continuous bi-directional carbon fiber composite, and (d) bi-directional fabric composite. The fiberglass mat layers were used to obtain a standard surface.

(drilled with a 2.5 mm-diameter wick), at 10 mm of the sample ends.

Electrical resistivity was determined by measuring the electrical resistance between the two contact points with a multimeter (Fluke, model 85), and then calculated as follows:

$$\rho = \frac{R \cdot A}{l_{ep}} \quad (1)$$

where R is the electrical resistance, A is the initial cross section of the specimen and l_{ep} is the length of the electrical path (distance between electrical contacts). This method, called the two-probe method, is not as reliable as the four-probe method, because the former involves the contribution of contact resistance. However, the two-prove method can be used for comparative measurements where the relative resistance change is been monitored, as it is done in the present work.

Tensile tests were carried out according to ASTM D-3039, with a constant displacement speed of 2 mm/min, in a universal testing machine MTS 311. Strain was measured with a contact extensometer (MTS 632.11F-20), until the deformation reached 0.3%. Mechanical properties, such as tensile modulus, tensile strength, and tensile elongation were determined, and compared with the results of a computer-assisted simulation (Composite Pro® software). After comparison, it was concluded that the fabricated laminates were good enough to be successfully associated to reliable test results, because its Young Modulus were in good agreement with those theoretically expected (see Table III). The theoretically calculated properties at fracture are lower than the experimentally calculated ones, and this has been attributed to micro-defects in the material (i.e., air bubbles, microcracks, etc.).

The relative change in electrical resistance was determined by recording the electrical resistance values every second during the tensile test, by mean of a multimeter (Fluke 198), and further calculated as follows:

TABLE II
Electrical Resistivity Values for Various Carbon Fibers Reinforced Composites with Different Fiber Type, Concentration, and Orientation

Reinforcement type	Concentration (% wt) – orientation	Fiber volume fraction	Electrical resistivity (Ω-m)
Chopped carbon fiber	2.5 – random	0.02	$(3 \pm 1) \cdot 10^{-2}$
	5 – random	0.04	$(1.3 \pm 0.4) \cdot 10^{-2}$
	10 – random	0.07	$(0.65 \pm 0.09) \cdot 10^{-2}$
	15 – random	0.11	$(0.5 \pm 0.1) \cdot 10^{-2}$
Continuous carbon fiber	31 – [0°]	0.24	$(1.3 \pm 0.5) \cdot 10^{-4}$
	31 – [90°]	0.24	$(2.5 \pm 0.7) \cdot 10^{-1}$
	31 – [0°/90°]	0.24	$(3.5 \pm 0.8) \cdot 10^{-4}$
Bi-directional fabric	31.7 – [0°/90°]	0.24	$(2.4 \pm 0.8) \cdot 10^{-4}$

TABLE III
Mechanical Properties for Various Carbon Fibers Reinforced Composites with Different Fiber Type, Concentration, and Orientation

Reinforcement type	Concentration (% wt) – orientation	Young modulus (GPa) Th.	Young modulus (GPa) Exp.	Tensile strength (MPa) Th.	Tensile strength (MPa) Exp.	Elongation to break (%) Th.	Elongation to break (%) Exp.
Chopped carbon fiber	2.5 – random	5.5	4.9 ± 0.1	–	40 ± 10	–	1.0 ± 0.2
	5 – random	7.1	5.7 ± 0.5	–	38 ± 7	–	0.7 ± 0.1
	10 – random	9.1	7.4 ± 0.6	–	39 ± 5	–	0.53 ± 0.05
	15 – random	13.5	9 ± 1	–	43 ± 8	–	0.5 ± 0.1
Continuous carbon fiber	31 – [0°]	57.8	64 ± 8	943.2	730 ± 6	1.6	1.36 ± 0.06
	31 – [90°]	6.7	5.7 ± 0.5	63.5	20 ± 7	1.0	0.4 ± 0.1
	31 – [0°/90°]	33.2	31 ± 1	475.4	340 ± 20	1.6	1.17 ± 0.07
Bi-directional fabric	31.7 – [0°/90°]	33.2	31 ± 5	483.8	280 ± 50	1.6	1.0 ± 0.1
None	–	3.86	3.68 ± 0.08	73.8	15 ± 2	2.5	0.41 ± 0.06

Th., theoretically estimated by employing Composite Pro® software; Ex., values experimentally determined.

$$\%\Delta R/R_0 = \frac{R_t - R_0}{R_0} \cdot 100\% \qquad (2)$$

where R_t is the electrical resistance measured at t time, and R_0 is the electrical resistance at $t = 0$.[1] These measurements were related to stress and strain values as a function of time, and then plotted against strain, to be compared with the stress–strain curve. The relative change in electrical resistance can be used instead of the relative change in resistivity, because according to previous reports,[1] the changes in cross section and electrical path are insignificant compared with the resistance changes because the deformation in composites is very small.

All digital photographs presented in this work were taken with a Sony Digital Camera FD Mavica® MVC-FD200.

RESULTS AND DISCUSSION

Electrical resistivity of composites

The electrical resistivity values for all composite arrangements are shown in Table II. All composites exhibited higher electrical resistivity than the fiber used (ρ = 1.72 · 10^{-5} Ω-m) because the conductive material is embedded in a dielectric matrix and, therefore, the conduction of electricity occurs mainly through carbon fiber basal planes (i.e., through the fiber axis).

The material with the lowest electrical resistivity is the one reinforced with unidirectional fibers tested at 0° because the conductive material (carbon fiber) is aligned with the testing axis, providing a very linear conductive path through the composite. The material with the next lowest resistivity value in Table II is the bi-directional fabric reinforced composite. The decrease in electrical conductivity (increase in resistivity) is due to the lower number of fibers aligned with the testing direction (0°), which is only half compared with the number in the unidirectional case, and only a small contribution from the weaved fibers in transversal direction (90°) is expected. Additionally, the cross-ply composite shows a slightly higher resistivity, since these transversely oriented fibers do not contribute significantly to the electrical conductivity. The transversely oriented fibers (90°) are separated from those fibers parallel to the testing axis by a resin (nonconductive) layer, and therefore their contribution to the composite electrical conductivity can be neglected.

The composites reinforced with chopped fibers exhibit a higher resistivity than the previously discussed materials, as shown in Table II. In these composites, the electricity is conducted by the pathway established by reinforcing fibers touching each other, being therefore a function of the fiber content. This mechanism also implies the presence of a threshold composition, under which there is no electrical conductivity along the overall composite, because the fibers do not form any continuous path through the material. The results presented in Table II show that the higher the fiber content the lower is the resistivity of the composite, and that all compositions studied are over the threshold composition for electrical conductance.

Finally, the composite with the highest resistivity value in Table II is the one reinforced with unidirectional fibers, tested in the transverse direction (90°). In this case, the electrical conductivity occurs through the occasional contacts between adjacent fibers, as previously reported by Kupke et al.[9] This contact is difficult due to the presence of "resin bridges" between the groups of fibers in the fabric, as illustrated with the photograph in Figure 2. These bridges tend to isolate the fiber tows from each other.

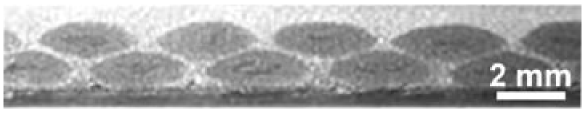

Figure 2 Digital photograph of unidirectional continuous carbon fiber reinforced epoxy, showing groups of carbon fiber and resin bridges between them.

Piezoresistive behavior in continuous fiber composites

Figure 3 shows a stress–strain curve and the relative change in electrical resistance with strain for continuous carbon fiber reinforced epoxy [0°]. The electrical resistance increases slowly with an increase in strain at low strain values, and it increases almost exponentially beyond 50% of the ultimate fracture strain. This increase in electrical resistance is caused by successive fiber fracture in the specimen during the test,[1] which indicates that the electrical resistance relative change measurement is a suitable way for monitoring damage in composites. It has been reported by Chung[1,6,7] that, for small strain values, a decrease in the electrical resistance relative change can be appreciated. This effect has been attributed to the alignment of the fibers in the direction of the applied load. However, this behavior was not observed by Kupke et al.,[9] or in the results presented here. After the (occasionally noticeable) initial alignment effect, the fibers fail progressively. The fracture of the fibers reduced the number of paths for electrical conductivity, and therefore the composite becomes a more resistive material, as observed in our tests. Progressive fiber failure occurs until the composite fractures catastrophically.

A similar plot to that presented in Figure 3 is shown in Figure 4 for continuous carbon reinforced epoxy [90°]. In this case, the electrical resistance increases gradually with strain up to 75% of the ultimate failure strain. At this point, an increase in the slope is appreciated, and then fracture occurs. The increase in electrical resistance is associated with fiber separation caused by strain, which diminishes current propagation through out the composite. At 75% of the ultimate fracture strain, what appears to be a single crack starts to grow in the matrix (as noted by visual observations and postfailure analysis), which produces the observed increase in the slope. That single crack, which is believed to be the first one generated, does not encounter any obstacle, and grows until composite failure occurs. The absence of other cracks in the material after the test, as shown in Figure 5, indicates that only the growth of a single macroscopic crack caused the catastrophic fracture of the composite.

For cross-ply [0°/90°] reinforced materials, the piezoelectric behavior is shown in Figure 6. Both this material and its mechanical behavior represent a combination among previously analyzed behaviors. The first increase in electrical resistance (until ca. $\varepsilon = 0.009$ mm/mm) is associated to transverse fibers separation when the matrix is deformed. This stage will last until transverse matrix cracks appear, as shown in Figure 7. Then, at higher strain values, the electrical resistance increases with a higher slope as a consequence of fracture of the fibers oriented in

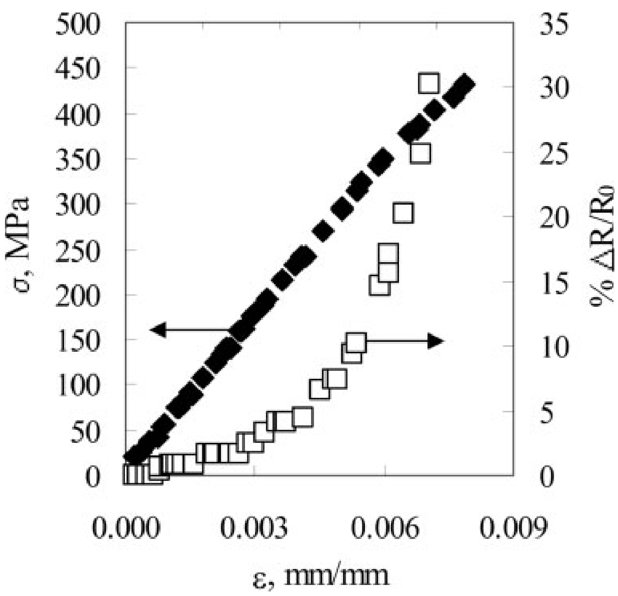

Figure 3 Variation of applied stress (◆) and relative change in electrical resistance (□) with strain, for a unidirectional continuous carbon fiber reinforced epoxy [0°].

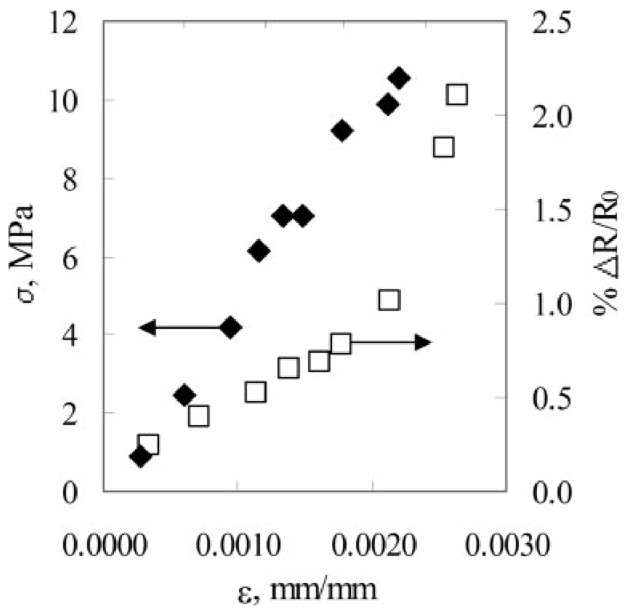

Figure 4 Variation of applied stress (◆) and relative change in electrical resistance (□) with strain, for a unidirectional continuous carbon fiber reinforced epoxy [90°].

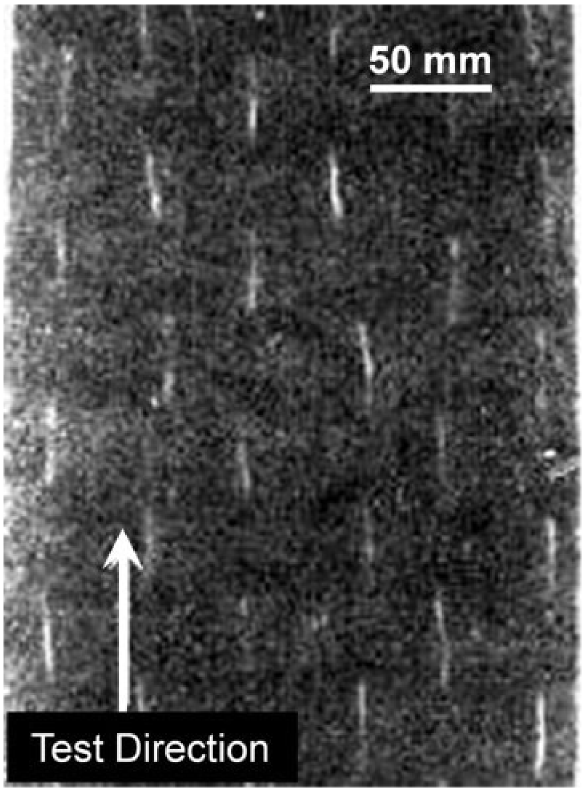

Figure 5 Digital photograph of a unidirectional continuous carbon fiber reinforced epoxy sample, submitted to tensile load in the fiber axis direction, showing the lack of cracks in the matrix. The white arrow indicates the test direction.

the direction parallel to the stress. However, it is difficult to determine when one stage finishes and the next begins. To do so, a simultaneous structural analysis during the tensile test would be required.

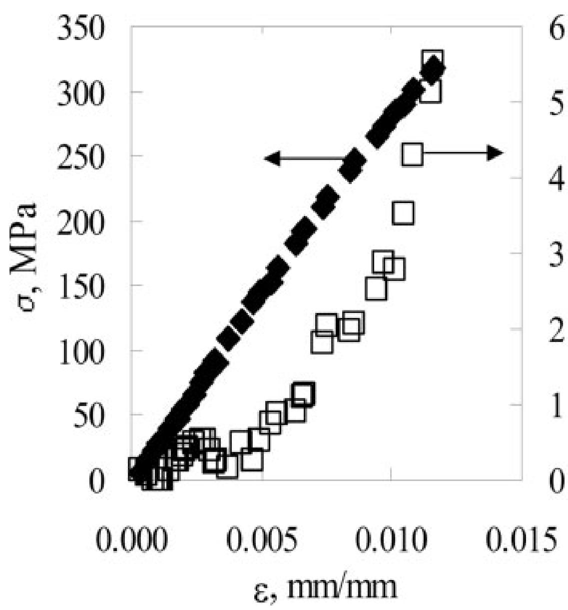

Figure 6 Variation of applied stress (◆) and relative change in electrical resistance (□) with strain, for a cross-ply [0°/90°] continuous carbon fiber reinforced epoxy.

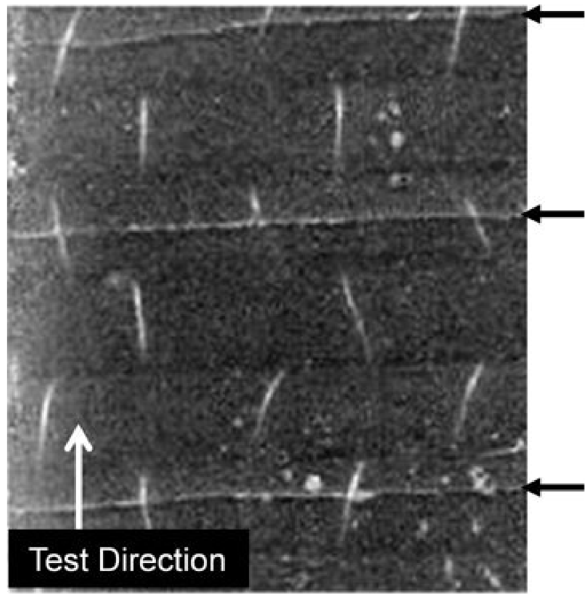

Figure 7 Digital photograph of a cross-ply continuous carbon fiber reinforced epoxy sample, submitted to tensile load in one of the fiber axis direction, showing the matrix cracks (black arrows). The white arrow indicates the test direction.

When the composite is reinforced with bi-directional fabric [0°/90°], a similar behavior to the cross-ply case was observed (Fig. 8). The initial electrical resistance increase is due to a separation of the transverse fiber upon composite deformation. At higher strain values, the fracture of parallel fibers produces higher resistance values, which are observed to increase in a slightly step-wise fashion. Kupke et al.[9] showed a similar behavior for a cross-

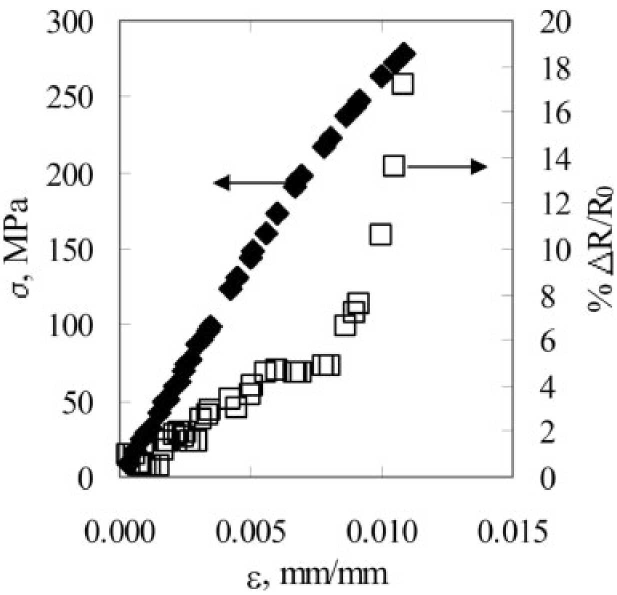

Figure 8 Variation of applied stress (◆) and relative change in electrical resistance (□) with strain, for a fabric [0°/90°] continuous carbon fiber reinforced epoxy.

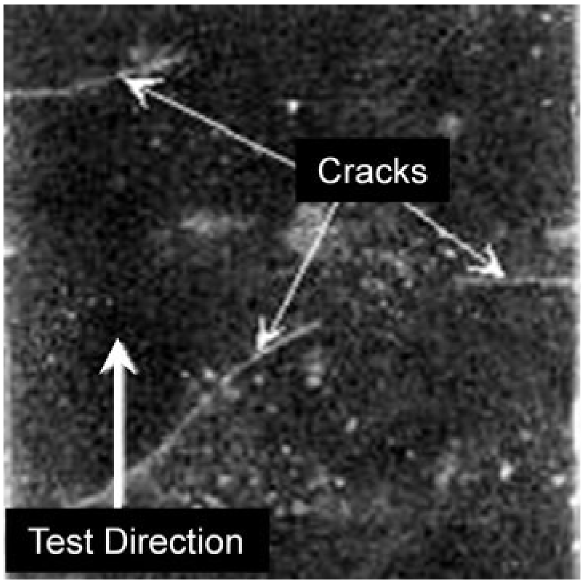

Figure 9 Digital photograph of a fabric continuous carbon fiber reinforced epoxy sample, submitted to tensile load in one of the fiber axis direction, showing transverse cracks in resin pockets. The white arrow indicates the test direction.

ply [0°$_2$/90°$_2$/0°$_2$/90°$_2$], from 60% fracture strain up to failure.

The reinforcement arrangement is different in each one of the continuous carbon fiber reinforced epoxy specimens analyzed in this work, as well as the corresponding piezoresistive behavior. Such differences may be the clue for the unique and rather different results presented here, and so far not discussed in previous works.

The presence of a "knee behavior" is a trend commonly found in the stress–strain curves for bi-directional reinforced composites. This phenomenon corresponds to the cracking of the matrix located among transverse oriented fibers.[10] Then, it should be expected that some variation in the electrical resistance behavior versus strain curve would occur. For instance, the step-wise increase shown in Figure 8 might be related to the generation of transverse cracks in the matrix. However, more evidence is necessary to support this claim.

If the step-wise increase was not observed in continuous fiber reinforced epoxy [90°], this is probably because one of the very first generated cracks will cause catastrophic fracture (check for absence of transverse cracks in Fig. 5). On the other hand, in a cross-ply laminate, the generation of multiple cracks is possible before catastrophic fracture, in those resin regions between parallel fibers, as it can be appreciated in Figure 7.

The cross-ply [0°/90°] composite presented here has lower mechanical resistance than that the cross-ply [0°$_2$/90°$_2$/0°$_2$/90°$_2$] composite presented by Kupke et al.,[9] since the former has a lower fiber content and is not a balanced laminate. Therefore, the cross-ply [0°/90°] composite will not be able to stand growing cracks as well as the cross-ply [0°$_2$/90°$_2$/0°$_2$/90°$_2$]. We assume that, the amount of transverse cracks in the first case is not enough to make the step-wise increase in the piezoresistive behavior detectable because the fracture occurs at an earlier stage than in the second case.

The bi-directional fabric reinforced composite has higher resistance to transverse cracks, because those cracks cannot grow through the complete cross section, and are only generated in resin pockets present throughout the material, as Figure 9 clearly shows. Therefore, these cracks can be generated, with a consequent sharp increase in the electrical resistance, without causing the failure of the composite. Then, it can be concluded that the modality in which the transverse cracks grow within a composite defines different piezoresistive behaviors.

Piezoresistive behavior in chopped fiber composites

The electromechanical behavior of chopped carbon fiber reinforced epoxy is shown in Figure 10, which corresponds to a composite reinforced with 2.5 wt % carbon fiber. The results obtained for different carbon fiber contents (5 wt %, 10 wt % and 15 wt %) were very similar to the presented one. Electrical resistance of composites increases during unidirectional tensile tests, as a consequence of the increase in chopped fiber separation due to matrix

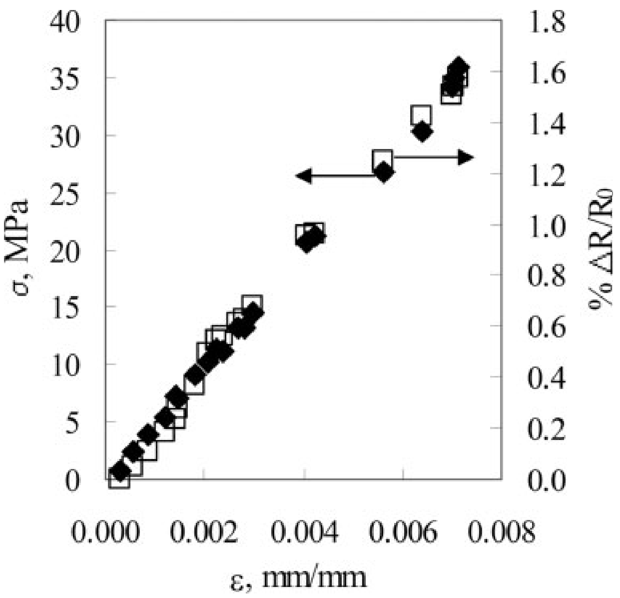

Figure 10 Variation of applied stress (◆) and relative change in electrical resistance (□) with strain, for a chopped carbon fiber reinforced epoxy with 2.5 wt % of carbon fiber.

deformation.[1,6,8] Therefore, in chopped fiber reinforced composites, the relative resistance change is a function of strain, instead of only a function of damage.

The relative change in electrical resistance as a function of strain, shown in Figure 10, is a straight line with a positive slope. This line is, despite the different scales, very similar to the straight line that corresponds to the stress–strain curve. The former observation leads to an important conclusion: the electrical resistance relative change can be associated to the tensile stress applied to the composite using the proper calibration factor. The previous feature allows the use of electrical resistance measurements as a tool for real-time strain monitoring of chopped carbon fiber reinforced epoxies. The stress applied onto a structure may be derived from the direct electrical measurement.

CONCLUSIONS

Carbon fiber reinforced epoxies can be used as self-monitoring materials for structures, as it was observed that a relative change in electrical resistance occurs when a tensile stress was applied. The electrical resistance relative change response can be used for monitoring strain in real time and, therefore, can also be used as a warning signal when the applied stress is getting close to the admissible maximum. The electromechanical response varies depending on the type and orientation of reinforcement in the laminate. Therefore, according to the specific requirements in applications involving smart structures, the reinforcement type should be chosen, not only considering its mechanical properties, but also its piezoresistive behavior.

For unidirectional continuous fiber reinforced epoxy, the electrical resistance relative change with strain presents an increase of electrical resistance in two stages, showing a change in slope. For chopped fiber reinforced epoxy, the behavior is completely linear and matches the stress–strain curve, indicating that this technique may be used for real-time strain monitoring. When the composite is reinforced with a bi-directional arrangement of continuous fibers, the $\%\Delta R/R_0$-strain curve shows what appears to be a step-wise increase. It was found that the generation and/or growth of matrix transversal cracks have a very important effect on the electrical resistance variation, producing sharp increases in this property as a function of strain. This phenomenon allows the use of real-time electrical resistance determination for damage monitoring.

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