RESISTIVE HEATING STRUCTURAL DAMAGE DETECTION IN NANOCOMPOSITES

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Keywords: Non-destructive Evaluation, Joule Effect, Carbon Nanofibers

1 General Introduction

Catastrophic structural failures are the cause of many physical and personal losses, and it is estimated that their prevention could save billions of dollars each year. Non-destructive evaluation (NDE) techniques [1], [2], [3] have been developed and employed for damage detection of structures such as planes, bridges, etc. to monitor cracks and other damages at pre-critical levels for remediation [4], [5]. Most of these techniques are however hard to implement in situ, so extensive research using new materials and technologies is highly needed.

Although approaches using carbon nanotubes (CNTs) for damage detection have been developed, they are mostly based on external coatings and thus require numerous electrodes, making it complicated to implement in a medium/large size piece [6], [7]. Recently, we have developed a novel approach based on carbon nanotube-resistive-heating effect that takes advantage of the effects that damages have on the electric and thermal transport in a material containing aligned carbon nanotubes [8], [9], [10]. When a difference of potential is applied to the CNT-containing composite laminate, the laminate is heated by Joule effect, also known as resistive heating. This heat is transmitted through the laminate causing “hot spots”. This heat flow is impeded in areas of damages [11] and these changes of temperature can be locally visualized with a thermal camera. The power operation depends on the amount of CNTs used, and we demonstrated a spatial resolution superior to the current state-of-the-art in non-destructive evaluation.

Despite recent efforts aimed at scaling up vertically aligned carbon nanotubes remains challenging within medium-size composite laminates. And because of this scaling limitation, most of the work using these nanoarchitectures was limited to coupon-size samples [13], [14].

In this work, we have implemented an approach using commercial grade carbon nanofibers (CNFs) mixed in a thermoset resin. Medium size plates (300x300mm²) can be fabricated using resin/CNF mixtures and conventional composites fabrication process, such as hand-lay up. In order to assess the resolution of this new approach, defects of different size and depth have been created in the carbon-fiber laminate. This laminate has been analyzed using c-scan and the resistive heating NDE technique.

2. Experimental

2.1 CNF/resin

A low viscosity polyester resin (Vipel F774-PTA, AOC resins) with 5% wt. carbon nanofibers (CNFs) supplied by Grupo Antolin Ingenieria was used as the composite matrix.

The carbon nanofibers used in this study were manufactured by Grupo Antolin Ingenieria using natural gas as a carbon feedstock, a Nickel compound as catalyst, and hydrogen and sulfur source at temperatures above 1100°C in a floating catalyst reactor. These commercial grade carbon nanofibers are formed by continuous helical-ribbon graphite layers. More information about these CNFs can be found elsewhere [15], [16]. Scanning electron microscopy has been performed to evaluate the CNF dispersion of the sample.
A rotational viscometer (Fungilab Alpha serie) was used to characterize the CNF/polyester resin.

2.2 Composite Fabrication

Two laminates, a thin (2 plies, 150x150mm²) and a thick (10 plies, 300x300mm²) were fabricated. Both laminates were made by hand lay-up using the polyester/CNF mixture as a matrix.

First, the glass fiber quadraxial fabric (QE 840, 840g/m², Mel Composites) was cut. Every layer was impregnated with the polyester/CNF mixture, accelerator and catalyst, following the manufacturer recommendations.

Thin polymeric squares were placed between the plies to simulate interlaminar defects in the thin laminate [19]. In order to evaluate the effect of the polymeric film thickness, films of different thickness were used: three-square defects were placed (2x2, 4x4, 8x8mm²) using a 0.175mm-thick PTFE film (Tooltec CS5 film, Airtech) and other three-square defects of the same dimensions were also placed using a PTFE film of 0.025mm in thickness (RF-242-R, Airtech).

To analyze the effects of different defect morphologies, the thick laminate was divided in three regions: a region without any damage, called “Non-damaged region”; and two regions with defects, “In-planted defect region” and “Hole defect region” (Figure 1).

Different shape defects (10x10mm² squares, 10mm-diameter circles, and 10x20mm² rectangles) made of glass fiber/PTFE film (0.13mm-thick, Merefsa S.L) were carefully placed between several plies of the laminate (1, 2; 3,4 and 5,6 plies) in the “In-planted defect region” (Table 1).

<table>
<thead>
<tr>
<th>Laminate</th>
<th>No. of plies</th>
<th>Dimensions (mm³)</th>
<th>Schematic layup</th>
</tr>
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<tbody>
<tr>
<td>Thin CF</td>
<td>2</td>
<td>150x150x1.3</td>
<td></td>
</tr>
<tr>
<td>Thick CF (In-planted defect region)</td>
<td>10</td>
<td>300x300x6.6</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Laminate details where black continuous line - glass fiber layer, grey discontinuous line – interlayer with defects

To assess the resolution of the technique, different size and depth flat-bottom hole defects [20] were drilled in the “Hole defect region” of the cured laminated (Figure 2).

Samples were cured during 5 hours at 100°C in a hot press. After curing, the specimens were trimmed using a diamond disc.

2.3 Non-Destructive Evaluation Experiment

In order to verify the position of the defects and the quality of the samples, the laminates were first inspected by ultrasonic c-scan, one of the most popular NDE techniques for composites materials.

Figure 1. Scheme of the 10-ply laminate and the defects created in the specimen for their detection through resistive heating NDE.

Figure 2. Scheme of the defects drilled in the “Hole defect region” of the 10-ply laminate. Bottom view of the surface of the sample.
A standard ultrasonic c-scan equipment for composite inspection was used (TecniTest Triton 1500 with an acquisition card type socomate USPC3100). For the thin and the thick laminate a 5MHz (IS-0203-HR) and a 10MHz (ISL-1003-HR) probe were used respectively.

Damaged specimens were heated via electrical current (Joule-effect heating) [8]. The temperature of the sample was recorded at 140x140 pixels, with a temperature range of -20°C to 250°C and a resolution of 0.1°C. In order to apply the current to the samples, silver-paint electrodes were applied on both ends of the laminate and were contacted by two wires by epoxy conductive paste. These two wires were connected to a power supply in order to heat the laminates. Because of the electrical resistance and size of the laminates, it took a couple of minutes to increase their temperature a few degrees over room temperature (~20°C), depending on the power applied. Once the sample was heated, thermal images were taken with a low-cost infrared camera (i7, FLIR).

3 Results and Discussion

3.1. Resin characterization

Because of the large surface area of the CNFs, the viscosity of the resin is around 4.5Pa at 20 rpm, which is a significantly high value for hand lay-up process. However, using the quadriaxial glass fiber fabric, we did not found any CNF filtering effect on the laminate during the impregnation or dry-spots. The CNFs dispersion has been evaluated by scanning electron microscopy (Figure 3.)

Analyzing the fracture surface of a CNF/resin sample, we have been able to detect small holes (<1µm diameter), probably created by the CNFs while expelled during the fracture process and short nanofibers distributed in the sample surface. Previous reports studied that these CNFs/CNTs dispersions are formed of clusters or agglomerates of CNF dispersed in the resin [17], [18].

In order to assess the quality of the laminates, ultrasonic c-scan analyses have been performed.

3.2. Non Destructive Evaluation: Ultrasonic c-scan

Using a conventional ultrasonic c-scan equipment, we could detect the 8x8mm² defects on the thin layer laminate. The damaged area appears larger for the thicker PTFE defects than for the thinner one (Figure 4).

Figure 3. Scanning electron micrograph of the fractured-surface of the cured CNF/resin matrix.

Figure 4. Two-ply laminate and the interlaminar defects created in the specimen (white squares). Top row corresponds to the interlaminar defects created with the thicker PTFE film and the bottom one to the thinner PTFE film (a). C-scan image of the two-ply laminate (b). The large green squares correspond to the defects placed in the composite.
For the 4x4mm² square defects, a small damaged region is seen by ultrasonic c-scan; however, this damaged region is not accurately defined as there are some other spots showing a similar pattern, such as the left corner of the laminate. There are also some small spots in the laminate distributed along the fiber direction, probably produced during the fabrication process. The 2x2mm² defects were not detected, neither for the thin nor the thick PTFE-simulated defects.

For the thick laminate, all the PTFE implanted defects were detected. However, despite the size of the defects, their morphology remained difficult to visualize.

Additionally, although the defects were introduced in different layers, it was not possible to identify in which layer they are located. From all the nine positions where defects were introduced, it seems that one has been moved during the laminate fabrication, as can be seen in the top-right corner of the c-scan image (Figure 5).

### 3.3 Non Destructive Evaluation: Resistive Heating

Once the samples were inspected by ultrasonic c-scan, silver paint electrodes were painted and wires attached to both samples, as described in the Experimental Section.

Resistive-heating-based non-destructive analysis was performed to both samples. First of all, the sample resistance was measured, in order to estimate the total voltage needed to heat the sample.

The resistance between the electrodes for the thin and thick laminate was of 250KOhm and 8.7KOhm, respectively.

In order to analyze the laminates fabricated, an electrical current (600V, 0.02A) was applied to the thin laminate. By monitoring the sample with a thermal camera, we observed that the laminate took less than a minute to increase its temperature by ∼5°C. Thermal images were taken every 30 seconds as in a conventional thermography inspection process. The infrared images revealed an inhomogeneous temperature distribution (Figure 6). However, this temperature distribution did not correspond with the defect pattern. The origin of this temperature gradients remains unclear, however, because of the number of defects detected in the laminate under c-scan and the inhomogeneous temperature distribution obtained on the sample, a deeper analysis using high-speed cameras synchronized with the power supply is needed.
For the thick laminate, as the defects are not placed symmetrically in the composite, the actual position of the thermal camera, which is imaging the non-drilled surface of the composites, is crucial (Figure 7).

![Infrared Camera](image)

**Figure 7.** Resistive-heating-based non-destructive evaluation system. The defects drilled on the laminate are placed on the opposite side of the thermal camera.

In order to heat the thick laminate, a power of 42W (600V, 0.07A) was applied. Because of the sample dimensions (300x300x6.6mm³), and physical characteristics of the sample (electrical resistance, thermal conductivity, etc.), it took several minutes to warm up the sample (Figure 8).

![Power Supply](image)

**Figure 8.** Ten-ply laminate (a). Temperature distribution of the sample heated using 42 Watts. The temperature distribution of the non-damaged region (top) has a different pattern than the damaged region (bottom) (b). The regions nearby the electrode reach temperatures as high as 80°C, with the highest temperature in the middle region of the electrodes.

Pictures were taken when temperature patterns were observed on the thermal camera. The heating process was repeated several times in order to get the clearest pattern. In the “non-damaged region” of the sample, the temperature pattern is symmetrical with respect to the middle of the sample. In the “Hole defect region”, small hot spots can be observed, corresponding to some of the drilled defects (Figure 9).

![Thermal Camera](image)

**Figure 9.** Bottom surface of the ten-ply laminate “drilled defect region” (a). Temperature distribution of the upper surface of the sample. Some clear blue dots can be seen on the thermography corresponding to the thinner flat defects of the laminate. Although the damaged area is just a few degrees over room temperature (blue region), the region nearby the electrode reach 80°C (red region) (b).

Using this detection method, we have been able to detect even the smallest defects introduced. However, defects up to 2mm in depth were not clearly appreciated. This might be due to the defect’s location (close to the electrode region) or the defect’s depth. It can also be noticed that the deepest defect drilled on the laminate are at higher temperature than the other ones, probably because of the thicker section of CNF/resin to be heated by Joule effect.
Interestingly, we also observed that we could not distinguish the size of the different defects. This is probably caused by the thermal conduction of the laminate, as previously reported in a study on the thermal conduction and the defect detection for lock-in thermography [20].

For the “Interlaminar defect region”, the temperature did not reach as high a temperature as the in the “non-damaged region”. Although the temperature distribution in the “Interlaminar defect region” is different than one of the “Non-damaged regions”, it was not possible to detect interlaminar defect. This different temperature distribution can be due to the effects of the “Hole defect region”.

4 Conclusions

We demonstrate here that resistive-heating-based NDE can be achieved using industrial scale carbon nanofibers dispersed in a composite matrix. For first time, medium scale laminates made of carbon nanofibers dispersed in the matrix have been evaluated by both c-scan and thermal resistive heating.

High-resolution damage detection has been demonstrated using the resistive heating NDE technique in composites. We were able to detect most of the defects generated in the panel (Figure 1), despite the small size of some of them (4mm of diameter). However, the resistive-heating-based NDE technique demonstrated here is sensitive to the type of defect. Although it seems a reliable technique for deep and small diameter defects, defects of up to 2mm depth cannot be detect using carbon nanofibers mixed in the resin and a low-cost thermal camera.

The main limitation of this technique is the high voltage it requires to heat the sample when carbon nanofibers are used as a conductive media in the composite matrix. Although the power needed is not particularly high (less than 50W for ~0.6cm³), it still takes several minutes to heat the sample. In order to evaluate these technique and compare it with commercial non-destructive evaluation techniques such as c-scan, high voltage power supply synchronized with high acquisition thermal camera needs to be implemented. Numerical analysis will also show the real potential of these techniques. Interestingly, several studies have been conducted in this field using carbon fiber, but there no study used electrical conductive nano-reinforcements [21], [22], [23].

Finally, commercial grade carbon nanofiber and non-resistive non-destructive evaluation can provide a new low-cost and effective inspection route for monitoring future generations of safer vehicles and infrastructure.

Acknowledgements

The NFRP project is partly funded by the European Commission under the 7th Framework Program, PCIG12-GA-2012-333924 (Marie Curie Career Integration Grant). The authors would like to thank C. Merino and E. Garcia from Grupo Antolin for supplying the resin mixed with carbon nanofibers. The authors would like to thank J. Vila, J.C. Rubalcaba and P. Romero for his assistance with the c-scan analysis, the thermograhy measurements, and scanning electron microscopy respectively. The authors also thank J.L. Jimenez for help and discussion regarding the sample fabrication. R.G.V. thanks S. Sanchez from University Carlos III Madrid for early discussions about the design of the drilled laminate. R.G.V. gratefully acknowledges the Spanish Ministry of Science and Innovation for financial funding through the Ramon y Cajal Fellowship.

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