

INTEGRATED STRUCTURAL HEALTH MONITORING OF LAMINATED COMPOSITES USING CARBON NANOTUBE YARNS UNDER STATIC LOADING: EXPERIMENTAL RESULTS AND VALIDATION

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Keywords: Carbon Nanotube Yarn, Piezoresistive Sensor, Integrated and Distributed Sensing, Damage Detection, Optical Fiber

ABSTRACT

This paper includes the experimental results of a study about localized damage detection in laminated polymeric composite plates using carbon nanotube (CNT) yarn sensors and validation using optical fiber sensors and x-ray tomography. A variety of CNT yarn sensor configurations were considered including CNT yarns stitched through the thickness of the composite laminate and CNT yarns placed straight cross-wide to the laminate. They were used to detect the presence of damage as well as its location, extent, and propagation pattern. As the laminated composite plates were mechanically loaded, the CNT yarn sensors captured instantaneously delamination and damage as demonstrated by their resistance history responses. In the cases where loading resulted in complete failure of the laminated composite, the resistance of the CNT yarn sensor was observed to rapidly increase to infinity. Crucially, the CNT yarn sensors are sensitive enough to provide a significant resistance increase output that serves as an indication of an impending delamination growth and propagation. The results from the CNT yarn sensors were corroborated by another in-situ experimental technique by using integrated optical fibers monitored through optical time-domain reflectometry (OTDR). In addition, post-mortem x-ray tomography imaging of the composite samples, were used to validate the experimental results. High performance optical fibers integrated in the laminated composite plates detect growing cracks in the composite sample when the damages reaches the optical fiber, allowing the OTDR to calculate the location of the damage along the optical fiber. A photodiode coupled with an amplifier were used to detect the time of the damage and correlate it with the CNT yarn sensors' data. Three-dimensional computerized tomography images captured the damage inside the sample.

1 INTRODUCTION

The need for more payload, increased speed, cost efficiency and resistance to varying environmental conditions have led to an increasing demand of hybrid structures in aerospace and civil applications. Composites due to their excellent properties like low weight, strength, stiffness,

resistance to corrosion, resistance to temperature, engineerability, and much more, have seen a leap in its usage in structural applications. However, with this growth, most structures are still reliant on traditional avionics and health monitoring techniques. Monitoring the structural health of composite materials and structures has been a challenge, due to their complex multiple modes and multi-stage process of failure. Unlike in traditional materials, damage in composite often initiates internally and could propagate through laminates undetected till catastrophic failure. The internal damage or failure could be caused by fiber breakage, matrix cracking, fiber-matrix separation or delamination. Some method of internal inspection is needed to identify small damage in composites. To ensure the structural integrity, non-destructive evaluation is utilized. Non-destructive evaluation (NDE) methods include acoustic monitoring, laser vibrometry, thermal imaging, piezoresistive carbon microfiber reinforcement, X-rays, and several other techniques [1]. They offer precise information regarding the structural integrity of a laminated composite but require costly, prolonged and complex procedures that make their use impractical in many applications. Structural health monitoring (SHM) methods provide constant and immediate feedback of the state of health of a structure including potential damage [2]. SHM methods may include vibration analysis, strain gauges, fiber optic sensors, stress wave propagation techniques as well as several other methods [2]. SHM methods that utilize micro-strain sensors can capture strain variation due to piezoresistive effects, resonance monitoring, piezoelectric effects, capacitance variation, or changes in optical properties [3].

Piezoresistive materials experience a change in their resistivity when subjected to a mechanical strain. Commonly used piezoresistive devices include metal alloy-polymeric films or metallic foil strain gauges and semiconductor strain gauges. Metallic foil strain gauges can capture very low fluctuations of strain with a maximum range of about 5% [3-4] while semiconductor strain gauges have higher gauge factors than metallic strain gauges. However, semiconductor strain gauges are sensitive to temperature limiting their efficiency. These piezoresistive strain gauges cannot detect initiating damage in composite materials with high compaction or multifaceted construction. There is also a form factor where these strain gauges can only be applied either on the surface or lack the aspect ratio to be integrated into complex structural components. More critically, they fail to achieve damage detection without altering the microstructure of the composite material. An alternative method of strain monitoring and damage detection that may offer the advantages of the previously mentioned methods without their drawbacks consists of the use carbon nanotube (CNT) yarns [5-9] that are intricately integrated into the fiber reinforcements of composite materials [10-14]. The concept is that CNT yarns are integrated in a laminated composite material forming a continuous sensor circuit, and their inherent piezoresistive sensitivity would capture small amounts of strain within the host material [10-13]. Unlike other SHM methods, integrated CNT yarn sensors may offer a non-destructive, simpler, easily customizable and robust alternative of damage detection in laminated composite materials.

In laminated composites, delamination occurs due to the separation of their layers mostly due to stress and matrix failure. Delamination represents a significant damage risk to their integrity [15] and can occur almost any place on the laminate; on the edge, near the surface, or at the center of the laminated composite. While the range in size of delamination and damage can vary drastically, it may elude the detection capabilities of most techniques that monitor change in material geometry such as metallic foil strain gauges, which are more suitable for surface strain detection, or optical fiber monitoring that requires complex equipment and data analysis. The ability of the CNT yarn sensors to detect mode II-dominated delamination in laminated composite materials had been previously shown by these authors [10]. The previous results obtained with the CNT yarn sensors were validated using two experimental techniques: integrated optical fibers monitored through optical time-domain reflectometry, and x-ray tomography of the entire laminated composite samples post-testing. High performance plastic multimode optical fibers were integrated in the laminated composite plates. The localization of damage was achieved through optical time-domain reflectometry (OTDR) and the time-dependence was monitored through a photodiode system. Once a growing crack or a delamination occurs within the composite sample, it damages or cracks the optical fiber as well, allowing OTDR to calculate the location of the damage along the length of the optical fiber. In addition, a photodiode with an amplifier was used to detect the time of the damage and correlate it with the CNT yarn sensors' data. Also, a three-dimensional computerized tomography (CT) imaging technique was used to corroborate the

damage in the laminated composite samples. These x-ray tomography images were not only able to capture the delamination and damage but could also detect the presence of CNT yarns inside the sample. Consequently, the results obtained with the CNT yarn sensors were corroborated by another in-situ experimental technique and by a post-testing imaging technique that specifically confirmed the damage location and its extent in the laminated composite. The results presented in this paper include an experimental study about the precise detection of delamination and minor damage including their exact location and extent using a combination of different types of integrated CNT yarn sensors. An attempt was made to provide a detailed explanation of the methodology and the mechanisms of capturing delamination using the CNT yarn sensors.

2 CARBON NANOTUBE YARN

The CNT yarns in this study were dry-spun from the sides of 400 to 500 μm -high vertically aligned arrays composed of multi-walled carbon nanotubes (MWCNTs) grown by water-assisted Chemical Vapor Deposition (CVD) [16-19]. The CNT yarns are composed of one or three intertwined threads. The CNT yarns composed of three threads have a total diameter of about 25 μm , a density of about 0.9 g cm^{-3} , and the twist of the single thread varies between 558 and 868 m^{-1} . A Scanning Electron Microscopy (SEM) image of the 3-thread yarn is presented in Fig. 1. The CNT yarns composed of one thread have a diameter of about 29 μm and a corresponding SEM image is shown in Fig. 1b. CNT yarns exhibit a piezoresistive response that depends on the exact construction of the material including the nanotube diameter, length and chirality, twist angle of the yarn, fabrication technique and other yarn characteristics [20,21]. The response of unconstrained 1-thread yarns subjected to quasi-static uniaxial tension loading exhibits a negative piezoresistivity [14]. The response of laterally constrained yarns is being determined and will be used to predict the response of the yarn sensors that are integrated in polymeric and composite materials. This piezoresistive characteristic of the CNT yarn is being used for sensing purposes and provide real time monitoring of the structural health of a polymeric or composite material through resistance measurements [10-13].

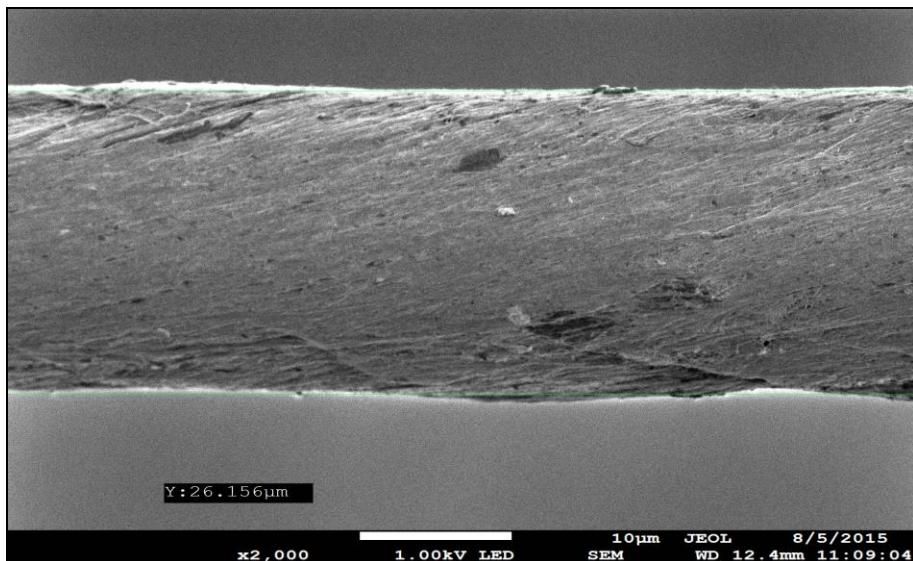


Figure 1: Scanning Electron Microscope images of CNT yarns.

3 DAMAGE DETECTION

3.1 Fabrication of self-sensing composite materials

The fabrication of the self-sensing laminated polymeric composite samples with the integrated CNT yarn sensors comprises three stages: CNT yarn integration into the dry fabric layers, curing of the laminated composites, and preparation of the yarn sensor circuits. It should be noted that, when

integrating CNT yarns into a carbon fiber laminated composite, the additional step of coating the CNT yarn is necessary to prevent short-circuiting between the carbon fibers and the CNT yarns; however, in this study, the CNT yarns are integrated in a glass fabric/epoxy laminated composite. The CNT yarn is secured to the head of a sewing needle and securely stitched into eight layers of the glass fabric, which constitute the central layers of the thirty-two-layer laminated composite. Before the first stitch is made, an artificial delamination is inserted between two of the central layers to control the location of delamination growth. After stitching the CNT yarns into the central layers, wires were connected to the extremities of the CNT yarn fibers using electrically conductive paint. Additional dry fabric layers are added to complete the layup of the laminated composite. A schematic of this self-sensing laminated composite sample, detailing the design of a sample containing various CNT yarn, is presented in Figure 2.

The next step in the composite fabrication process consists of the impregnation of the yarn sensor-integrated composite layers using a hand lay-up process. The polymeric phase is the commercial product ToolFusion™, a room temperature-curing epoxy system composed of resin and hardener, which are combined in a 5:1 resin to hardener ratio. After impregnation, the layers are sandwiched between additional layers of dry carbon fiber layers, acting as bleeders to maintain an approximately 65% fiber volume fraction. The composite sample is then placed in a mold and cured under full vacuum and 480 kPa of pressure for six hours. After the self-sensing composite sample is removed from the mold, the wires and contacts are rechecked to ensure they are intact.

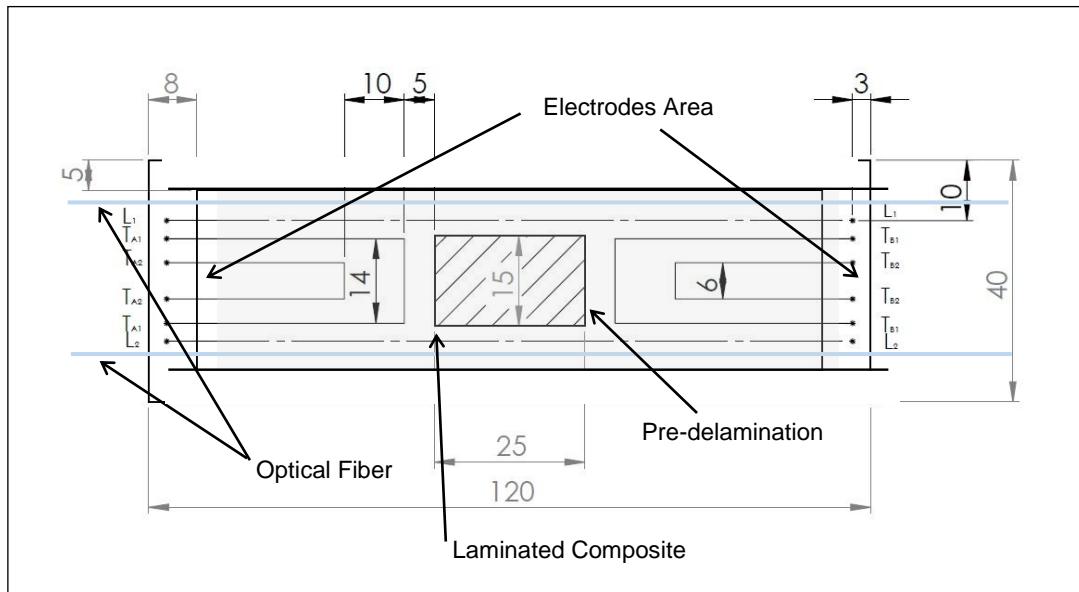


Figure 2: Schematic of a 32-layer glass/epoxy laminated composite of the integrated yarn sensors including stitched ones (through layers 12-21) and straight ones (between layers 16 and 18), with a 25 x 15 mm central delamination. Wires are later connected to the CNT yarns for resistance measurements.

3.2 Coupled mechanical and electrical measurements

The coupled mechanical and electrical characterization of the self-sensing composite samples requires accurate and sensitive testing equipment to capture load, displacement and resistance histories. An MTS Criterion 43, displacement-controlled mechanical testing platform fitted with a 30 kN-load cell, is used in combination with a 3-point bending fixture as shown in the schematic of Fig.3.

Loading is monitored by a Testworks 4 software controller that acquires load and displacement histories data at 1 Hz. To minimize the effect of the contact resistance, the two lower supports are positioned between the electrical connections of the stitched yarn sensor circuits, thus creating a zero-stress area near the electrodes throughout the entire loading process. The self-sensing composite samples are loaded at a rate of 0.2 mm min⁻¹.

Electrical monitoring of the self-sensing composite samples requires a measurement system that can accommodate to the differences in the resistance ranges of each yarn sensor circuit. A National Instruments (NI) data acquisition chassis (DAQ) Model 9178 equipped with an NI 9219 card is used along with an NI 4072 digital multi-meter (DMM) mounted on an NI PXI 1033 chassis. The DMM can monitor a single yarn sensor circuit at a time with a maximum resistance range of 1 MΩ, while the DAQ chassis can capture multiple yarn sensor circuits with a maximum range of 10.5 kΩ. The resistance monitoring is achieved with a NI LabVIEW Signal Express software program. Electrical connections to the self-sensing composite sample secured in the 3-point bending fixture are made using small alligator clips. The resistance history data is acquired at a rate of 1 Hz.

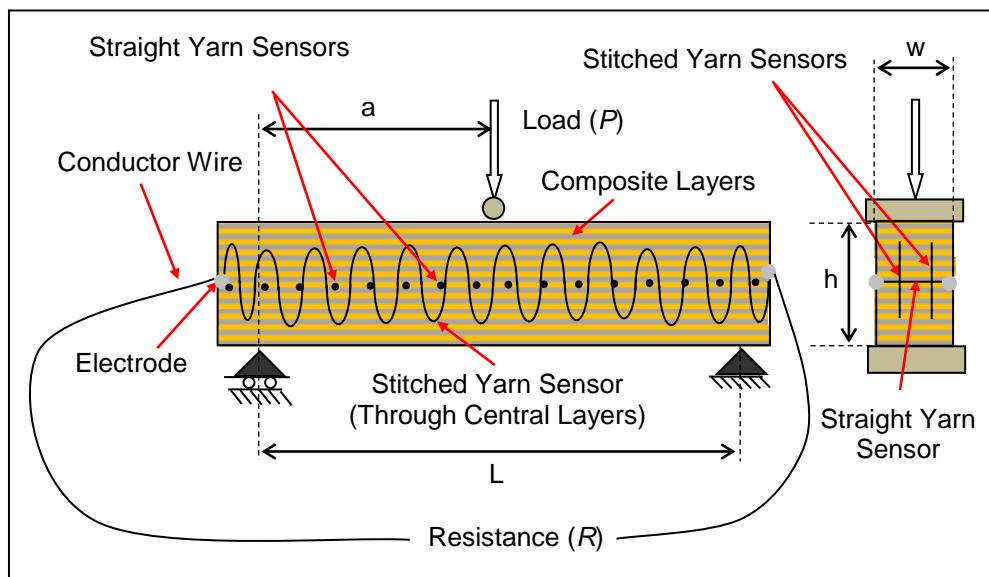


Figure 3: Schematic of experimental setup of self-sensing composite sample subjected to 3-point bending: side and end cross-sectional views of laminated composite beam sample instrumented with stitched and straight yarn sensors [13].

3.3 Damage detection using CNT yarns

CNT yarn sensors integrated in laminated composite materials have been shown capable of delamination detection [10]. The mechanical/electrical response of self-sensing composite samples with a glass plain-weave architecture and combined sensor configurations is presented in Figure 4. Figure 4 shows the load, P , in terms of time, t , representing the load history; and the resistance change, ΔR , or the difference between the actual resistance and the initial resistance in terms of time, representing the resistance history. Delamination in the sample is identified by the sudden decrease of the maximum load in the load history curve (event A). Delamination is detected by the stitched yarn sensors as evidenced by the increase of the resistance to infinity (events B1/B2). The time difference between events B1/B2 and A was 214 s. This difference implies that the yarn sensor can capture the delamination instantaneously. This response of the yarn sensor demonstrates its ability to not only

detect the delamination but also to anticipate it (events B1/B2) even before the load-history response of the laminated composite sample indicates it (event A). In addition, the yarn sensor could withstand loading more than the maximum load as well as capture the delamination without its circuit failing. The yarn sensor failed (event C1) 94 s after event A as shown in Figure 4.

The determination of the exact location of delamination and its progression can be achieved with a configuration consisting of a combination of different yarn sensors like the one shown in Figure 2, which includes stitched yarn sensors and transverse yarn sensors. The yarn sensors stitched through the thickness of the laminates allows for the determination of delamination only; additional transverse yarn sensors parallel to the composite laminate layers and along the beam's width direction are required to establish the precise location of the delamination or the damage. Figure 2 exhibits a schematic of a fabricated self-sensing composite sample containing both types of yarn sensors. It is worth mentioning that damage detection based on a significant resistance increase does not require highly precise resistance measurements and thus two probe measurements are deemed appropriate and sufficient. Figure 4 shows the load and resistance histories of each yarn sensor.

An artificial rectangular delamination of 15 x 25 mm was centered between the two stitched yarn sensors allowing a gap of 2.5 mm between the yarn sensors and the delamination [13]. Figure 4 shows the load and the resistance change histories of the two stitched yarn sensors (#1 and #2). It is observed that all the previously described events are also present and some occur almost simultaneously (time delay of 38 s between events B2 and B1). Both yarn sensors withstood the applied load and responded in a similar way demonstrating a relatively symmetrical growth progression of the initial delamination.

The two stitched yarn sensors in Figure 4, indicated as continuous and dotted red lines, respectively, show sensitivity to the deformation throughout the entire loading process of the sample as indicated by the resistance increase. Both detect the delamination as shown by the corresponding jumps in the resistance and the time delay between these events (B1 and B2) is 38 s. This time delay could be attributed to a slight asymmetric growth of the initial delamination.

In these experiments, four transverse yarn sensors were placed, as indicated in the inset of Figure 4. The transverse/longitudinal yarn sensor on the left side closer to the delamination, indicated as a continuous brown line (transverse/longitudinal yarn sensor circuit #5) in Figure 4, exhibit an increasing resistance of few ohms over time. This transverse/longitudinal yarn sensor does not fail because the delamination does not reach it before the sample experienced a significant deformation and the test is stopped. The wire of the sensor on the left side closer to the delamination, indicated as a continuous purple line (transverse sensor circuit #6) could not be analyzed, because the wire connected on this sensor was lost before the beginning of the test. However, as sensor #5 was closer to the delamination and did not fail, it can be assumed that sensor circuit #6 did not fail either, given it was further from the delamination than sensor circuit #5.

Transverse sensor circuit #3, indicated as a continuous black line in Figure 4, exhibits a resistance decrease of about 0.36Ω before failing when the delamination reaches its location about 304 s after the delamination reaches the first stitched yarn sensor. Transverse sensor circuit #4, indicated as a continuous green line in Figure 4, failed right after sensor #3, exhibiting a resistance decrease of about 0.35Ω before failing at about 374 s after the delamination reached the first stitched yarn sensor and 70 s after the delamination reached sensor circuit #3. Many experiments were run using shorter beam configurations and it was observed consistently that the yarn sensor closest to the delamination fails first and subsequently the other transverse sensors fail as the delamination propagates and reaches their locations.

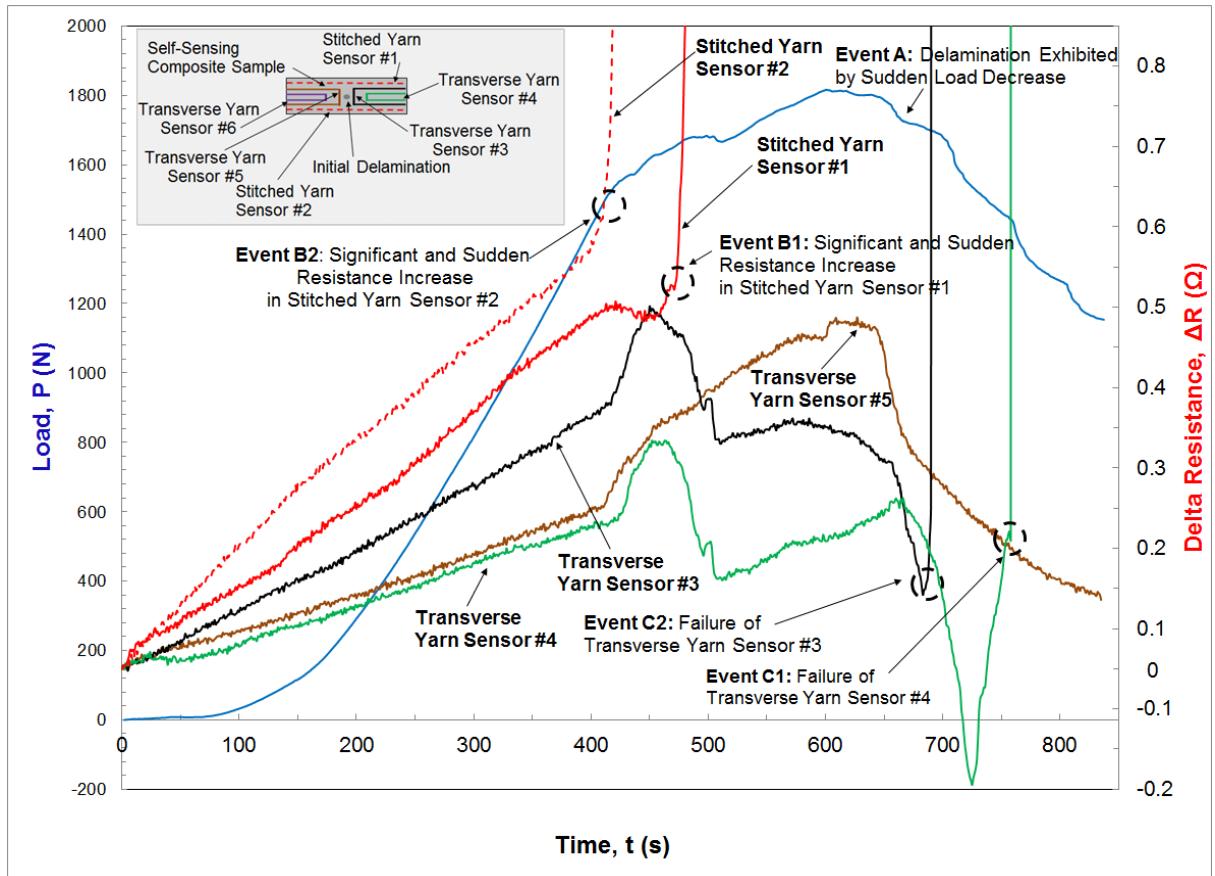


Figure 4: Localized detection of major delamination in 32-layer glass/epoxy composite sample using a combined stitched and transverse/longitudinal yarn sensors configuration: load and resistance change versus time curves.

4 VALIDATION OF PREVIOUS RESULTS

The previous results obtained with the CNT yarn sensors were validated using two experimental techniques: integrated optical fibers monitored through time-domain reflectometry and x-ray tomography of the entire samples.

4.1 Optical fiber sensors

Fiber optic technology has been used for communication purposes for decades. The main structure of a fiber optic cable is composed of the core, the cladding and the coating. The core is the component that transmits the light along the fiber optic cable. The cladding surrounds the core and creates the “total internal reflection” phenomenon to keep the transmitted light within the core. The coating protects the internal elements of the fiber optic cable from environmental effects.

There are different types of sensors based on the optical fibers. The simultaneous validation of the CNT yarn sensor was performed by using optical time domain reflectometry. OTDR is generally used for characterizing the light transmission properties of the optical fibers by sending very high-speed light pulses and analyzing the reflections from possible defects (e.g. splice points, connectors, cracks etc.). The location of the defects and the optical losses caused by them can be easily determined by modern OTDR equipment [22]. The v-OTDR system (i.e. Luciol Instruments v-OTDR) used in this experiment detects the number of photons that are reflected from any kind of reflection point within the optical fiber. The system uses 651.5 nm-wavelength laser pulses through the optical fiber and

attenuates the back-reflected light. Only one photon comes back for each pulse because of the attenuation process. The system counts the detected photon number and scans the delays to analyze the backscatter of the entire fiber and identify the location of the reflection surface [23].

In the scope of this study, the v-OTDR device is used for locating the damage along a 250 μm -diameter multimode plastic optical fiber as seen in Figure 5a. Once a growing crack or a delamination occurs within the composite sample, it damages or cracks the optical fiber as well; which allows the v-OTDR to calculate the location of the damage along the optical fiber. Thus, it is possible to pinpoint the damage around an optical fiber within the composite specimen during the experiments.

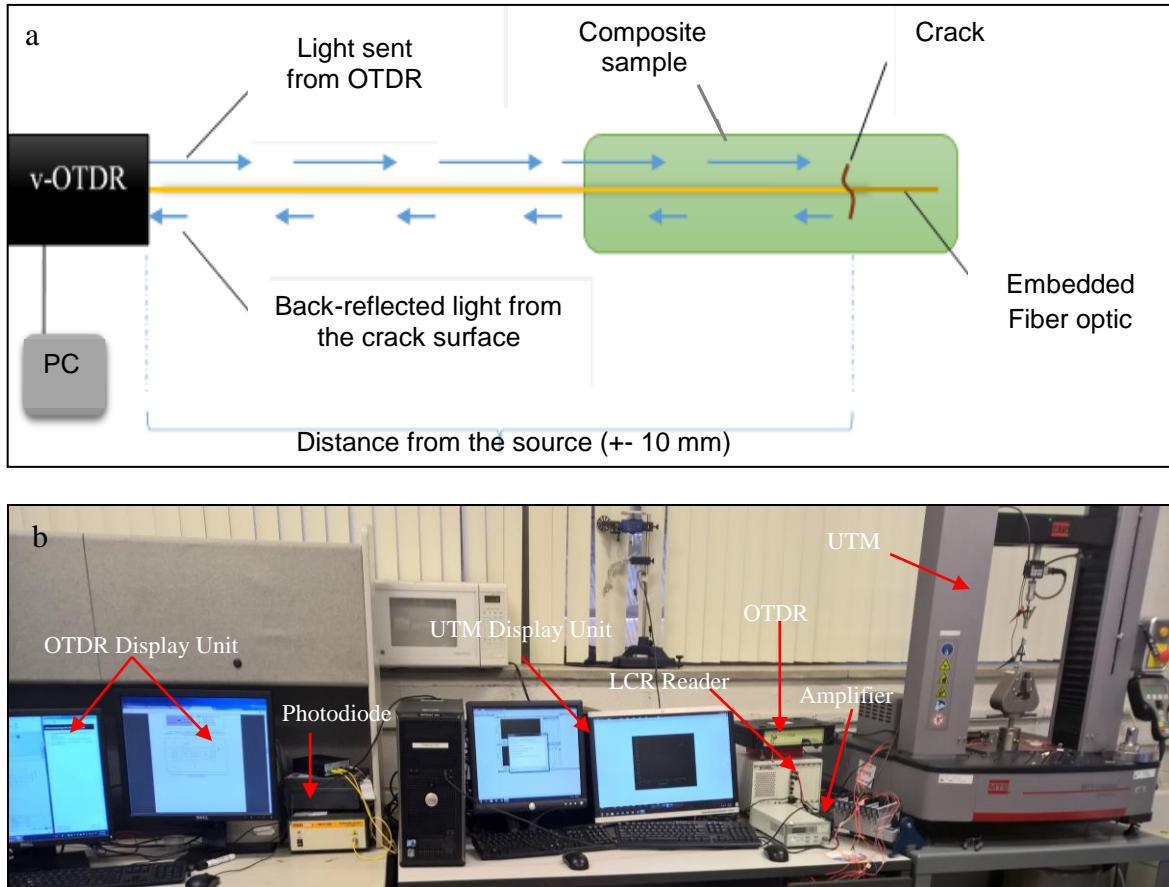


Figure 5. (a) Schematic of v-OTDR crack detection process. (b) Optical image of the experimental setup including the OTDR and an MTS Universal Testing Machine (UTM).

During the manufacturing process, two bare multimode optical fibers with SC connectors were located parallel to the stitched CNT yarns along the axial direction of the specimen. The inlet of the fiber optic cables was supported by the plastic jacket to prevent any possible failure at these critical locations during the handling of the specimen. The tips of the fiber optic cables were coated by a silver paint to increase the reflectivity for better determination of the endpoint locations using v-OTDR. Prior to the experiment, the initial characteristics of the embedded bare fiber optic cables were observed and recorded by using the v-OTDR equipment. Right after that, the experiment was started and the v-OTDR data was recorded for one of the fibers in conjunction with the CNT yarn sensor data. The response of the CNT yarn sensor at certain points of the experiment was compared with the v-OTDR data for validation purposes. The other optical fiber was tested using v-OTDR after the experiment to locate possible damage locations.

In using the OTDR, the peak measurement of value identifies a reflective event which is then measured and the reflectance calculated. The OTDR is programmed by manufacturers with the refractive index of the fiber to make it possible for the OTDR to calculate the length and position of the test events defined by the reflected or backscattered light as the measurement pulse travels along the fiber length. Higher peaks indicate higher reflectance. In Figure 6, the thick blue lines represent the length of the fiber optic before the delamination test. At around 0.2 meters, there was a sharp peak in back-reflection indicating the occurrence of an event. The CNT yarn sensor around the location of this reflection was confirmed to have failed through delamination. This means that when the delamination reaches both the CNT yarn and fiber optic, they can be captured simultaneously by both sensors. To confirm if the optic fiber itself failed or was just hit by the propagating delamination, post-test imaging would be needed. However, plastic optical fibers can withstand more strain than the CNT yarn sensor without failing. Since it would be ideal to monitor both sensors to failure, a less flexible optical fiber is needed. Therefore, glass optical fibers could be more suitable to this application due to its delicate nature and higher information transmission capacity with lower loss. The use of glass optical fibers in this application is currently under study.

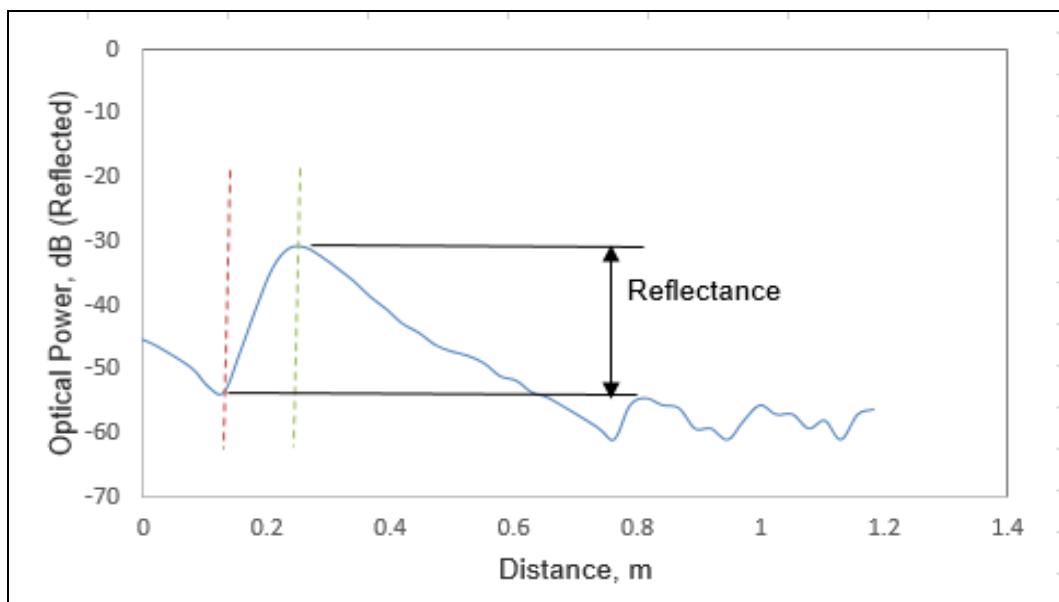


Figure 6. Reflection versus distance curve of the fiber optics cable.

4.2 X-ray tomography

To validate the experimental results, the sample, after tested, was analyzed using computerized tomography (CT). Tomography is an x-ray system able to identify core damage characteristics in a material, such as type, exactly location, geometry, and orientation. The basic principle of tomography is to place the object that will be tested between a combination of two x-rays, a ray source and a ray detector. The rays emitted by the source travel through the object until the detector where the total absorption is retained and measured by the ray. Thus, through the measurements provided by the x-ray, a computer system can generate images from the object. The method is feasible for damage detection in composite samples due to its high image technology results. There are several available types of mechanisms capable of performing CT in composites. These experiments utilize a CT system that combines x-ray images from different angles and planes and covert them into a single image that represents a cross-section view of the specimen. The results from the tomography system in damage detection of our sample, are images able to quantify and identify exact location of the failures. The resolution of roughly 0.020 mm is high enough to our sample to correlate with the data from the mechanical testing machine. Some of the results obtained on a composite sample are shown in Figure 7.

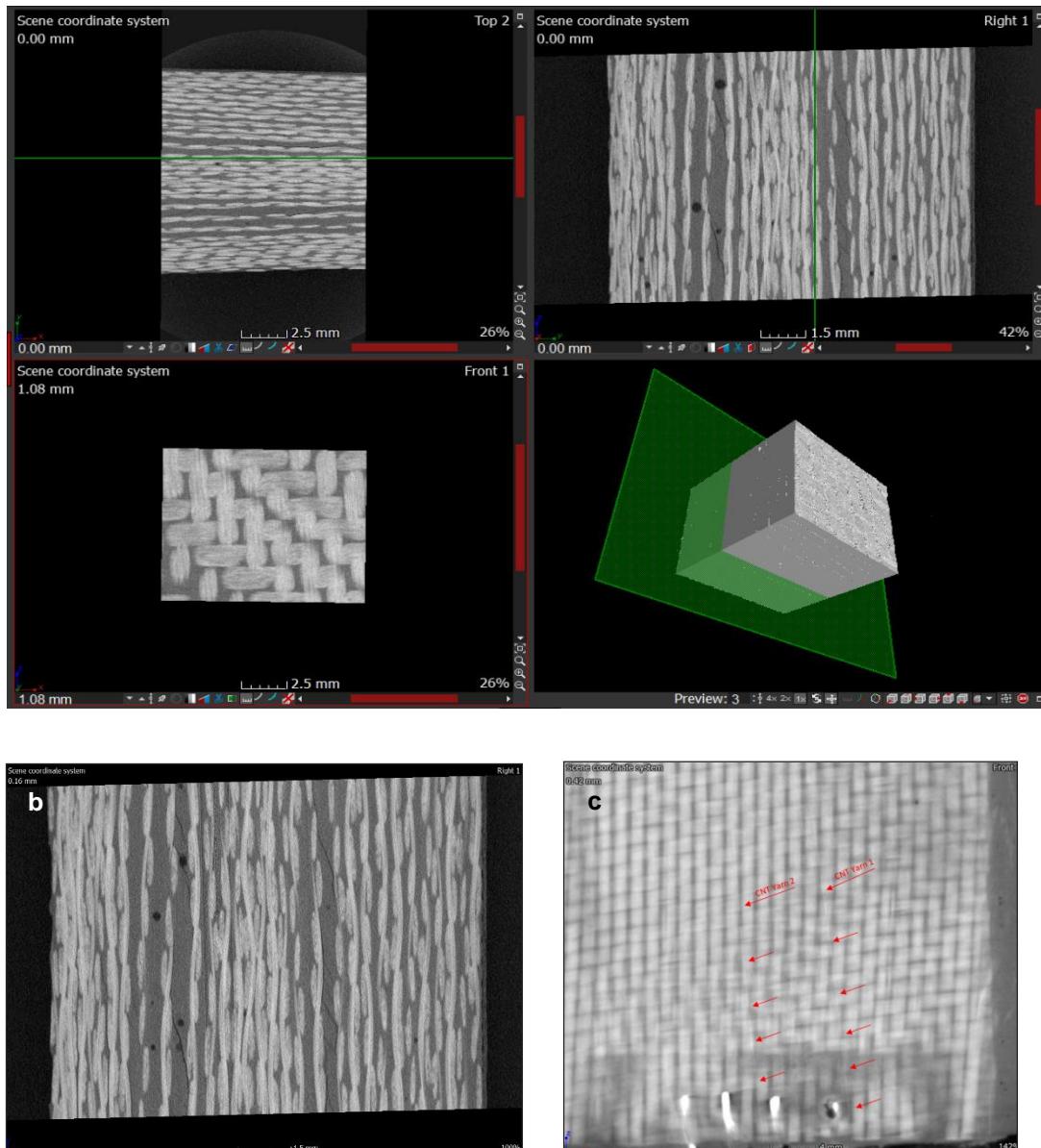


Figure 7: X-ray tomography images of the self-sensing composite sample tested: (a) different cross-section cuts and perspective view. (b) Cross-section showing double delamination. (b) Transverse CNT yarns.

5 CONCLUSIONS

An experimental study was conducted to evaluate the ability of piezoresistive-based, integrated and distributed, carbon nanotube yarn sensors to detect delamination in laminated composite materials, determine the specific location of the damage and delamination, and further validate the results using different sensing approaches. The study included monitoring the growth of a preset delamination defect during loading of glass fabric polymeric laminated composite samples using integrated yarn sensors. The study showed the ability of the yarn sensors to not only capture the delamination but also anticipate it as exhibited by a significant increase in the resistance of the stitched yarn sensors ahead of the delamination. The exact location and progression of a delamination was determined by additional transverse yarn sensors that yield a higher resistance output when the delamination reaches their specific locations. All the previous findings further contribute to demonstrate the feasibility of a multi-sensor network of integrated and distributed carbon nanotube yarn sensors to monitor an entire designated area and pinpoint the exact location of damage. These carbon nanotube yarn sensors provide excellent piezoresistive response to loading without compromising the integrity of the laminated composite, offering thus the potential for developing a highly adaptive, practical, and sensitive structural health monitoring method. The validation of the results with the CNT yarns was conducted using optical fiber sensors and x-ray tomography.

ACKNOWLEDGEMENTS

The authors thank all the students in the Intelligent Materials laboratory at The Catholic University of America (CUA) for their support to this research project. The authors also thank the financial support from the Air Force Office of Scientific Research (AFOSR) through Grants FA9550-10-1-0040 and FA9550-15-1-0177, with program officers Dr. David Stargel, Dr. James Fillerup and Lt. Col. Jamie Morrison, and from the National Aeronautics Space Administration (NASA) District of Columbia Space Grant Consortium (DCSGC) through Grants 31154 and 31377. The authors also thank Brazil's federal agency, CAPES, for providing the financial support to all the Brazilian students to conduct studies and research at The Catholic University of America.

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