

MULTI-STAGE FATIGUE LIFE MONITORING ON CARBON FIBRE REINFORCED POLYMERS ENHANCED WITH MULTI-WALL CARBON NANOTUBES

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ABSTRACT

The non-destructive monitoring of the damage developed in carbon fiber reinforced polymers (CFRPs) during mechanical loading is a key issue in many applications, especially in aerospace structures. Previous studies during the last decade have shown that the mechanical deformation and the electric resistance of CFRPs are closely connected, so that the material can act an inherent sensor of his own damage. In such composites with continuous carbon fibers, fiber breakage causes sudden increase in the axial resistance and in this way damage sensing is possible. Carbon nanotubes (CNTs) are one of the most promising mechanical reinforcing materials for polymeric composites due to their ultra high axial Young's modulus and strength, high aspect ratio, extremely large surface area, and excellent thermal and electrical properties. In this study, CNTs were used as modifiers of the epoxy matrix of quasi-isotropic carbon fibre reinforced laminates. In the present studies the dual role of the nanotubes was investigated: first as reinforcement of the matrix materials and second, as electrical sensors for damage monitoring. The prepared laminates were subjected to tensile monotonic loading and tension-tension fatigue and, the changes in the longitudinal resistance were monitored via a digital multimeter. In addition, the Acoustic Emission and Acousto-Ultrasonic (AE/AU) techniques were used for monitoring the fatigue process of the laminates and all the above data were correlated with data taken from a Thermal camera monitoring device.

1. INTRODUCTION

Fiber reinforced polymer (FRPs) materials and especially Carbon Fiber Reinforced Polymers (CFRPs) are widely used in aerospace applications. Due to their high strength and stiffness combined with lightness, CFRPs comprise better design choice over typical metallic structures. Additionally multi-functionality is an aspect that aerospace technology is focusing on the last decades. Design parameters like the mass reduction with increased system efficiency demand multifunctional approaches. The technology concept of multifunctional materials with sensing capabilities combined with enhanced mechanical-electrical and/or thermal properties could prove useful for the high requirements of the aerospace sector.

Continuous health monitoring of composite materials and structures can contribute to enhance safety and minimize cost by optimizing maintenance protocols of structures. Acceptable health monitoring sensors should meet specific requirements such as small weight and size, high sensitivity, structural compatibility in the case of built in sensors, life long operation capacity, ability for health monitoring of large critical areas of the structures and possibility to transmit information to a central processor in real time [1]. The optimal way to proceed for structural health monitoring is to use the material itself as sensor. To this direction previous studies have employed electric resistance change (ERC) method to identify internal damage of CFRPs under mechanical loading [1-11]. It has been proven that in the case of CFRPs the electric resistance, the mechanical

deformation and damage are closely connected, so that the material can act an inherent sensor of its own damage. Additionally, electric conductivity in the longitudinal direction exists due to the inherent conductivity of the carbon fibre, while transverse electric conductivity is realized by contacts between the neighboring fibres and plies. The electric resistance change method does not require expensive equipment for instrumentation. Since the method adopts the conductive elements of the CFRPs as sensors for damage detection, this method does not cause any deterioration of mechanical performance both under quasi-static or fatigue loading conditions and the method is directly applicable also to already existing structures. Furthermore, the ERC method does not increase the structural weight. However until recently all those significant advantages of the ERC technique were outflanked due to its medium sensitivity that inhibited the detection of the damage at early stages when still no macroscopic catastrophic phenomena take place.

The introduction of nanotechnology in the field of composites with nano-scaled fillers, such as carbon nanotubes (CNT) offered new possibilities towards this direction. Taking into consideration their high axial Young's modulus, high aspect ratio, large surface area, and excellent thermal and electrical properties [12] these fillers can be used as modifiers for the polymer matrices of the CFRPs leading among others to a significant increase of electrical conductivity of the epoxy matrix [13-15], while a good correlation between fatigue damage and increase in resistance has been also determined [16].

CNT are molecules of carbon related to two other carbon crystal forms, graphite and diamonds [17]. They are often described as looking like rolls of graphite chicken wire, but CNT are actually part of the fullerene family; they are essentially buckyballs expanded from the center into cylinders. Today, there are around 44 producers worldwide ranging from single walled nanotubes to nanofibres. All the types of CNTs are produced mainly by three techniques: arc-discharge, laser-ablation, and catalytic growth. The CNT production has reached a tipping point where the combination of decreasing prices and increased availability will enable more widespread applications [18].

The present study comprises an investigation of the capacity of the CNTs to be used as inherent sensors utilizing an improved and more sensitive electric resistance change method for common CFRP composites. In addition the overall mechanical and electrical performance of the CNT modified composites is investigated and compared with the performance of reference composites. At the beginning unidirectional composites with various CNT contents and reference polymer resin matrix were used for quasi-static tensile and cyclic loading-unloading-reloading tests, to show that matrix damage at relatively low strain level causes detectable variation in the composites resistance and to investigate systematically the electromechanical behaviour versus the CNTs content. Moreover the effect of the nanodopants on the fracture behaviour of the laminates is studied. In second phase, quasi-isotropic composites were used in order to quantify the CNT doping effect during tests that approximate increased real service life conditions. Tension-tension fatigue and low velocity impact tests are selected in this case. Especially during the tension-tension fatigue tests the longitudinal resistance was monitored in order to establish a link between the presence of CNTs with the damage accumulation.

2. EXPERIMENTAL

MWCNTs produced by catalysed CVD, were supplied by ARKEMA, France. Their diameters were 10-15 nm and they were more than 500 nm long. The nanotubes were dried in an oven overnight prior to use. The epoxy system used for the fabrication of CNT doped resins was the Araldite LY564/ Aradur HY2954 from Huntsman Advanced Materials, Switzerland. The dispersion of the MWCNTs in the epoxy took place in a Torus Mill device (VMA Getzmann GmbH). The torus mill introduces high shear forces by a high-speed rotating disc and reduces the nanoparticle agglomerates due to the milling effect generated by zirconium dioxide beads. The beads have a diameter of 1.2 to 1.7 mm and cause strong shear action and collision effects. The dissolver-disc provides additional shear forces and maintains the vortex flow. The compound is stirred in a vacuum container to avoid air inclusion. The vortex flow achieved by the geometry of the disc leads to continuous mixing of the compound. The mixing speed was at 2000 rpm for 3 h and the CNT content was 0.5 %. The manufacturing of CFRPs with the above resin as matrix material was the next step. The Carbon Fibre (CF) laminas were chosen to be quasi-isotropic [0, +45, 90,-45] s of 16 plies by Wela, Germany with weight of 160 gr/m². Each panel was hand laid-up and then processed in an autoclave, using the vacuum bag technique. A reference panel was also manufactured with neat resin for direct comparison. Prior to fatigue, tensile test performed in order to investigate the influence of the CNTs and to collect max stress values. The fatigue parameters were $f=5$ Hz, $R=0.1$ and stress levels of 80%, 70% and 60% of maximum stress were used. The electrodes were placed on top and bottom edges of the specimens using silver paint and silver tape and the resistance changes were monitored by a Keithley Digital Multimeter. As for the AE/AU techniques the sensor were placed in the following manner; the pulser at the centre of the specimen and the two receivers 60 mm apart the pulser.

3. RESULTS AND DISCUSSION

In Fig. 1 the change of the longitudinal electric volume resistance is presented versus strain during monotonic tensile loading up to 11 kN estimated to be the 50% of the maximum bearing load for certain composite layout. For this type of CFRP the resistance seems to follow the linear trend of the load but no significant difference is observed comparing the doped and neat specimen. This phenomenon is mainly attributed to the quasi-isotropic layout that creates multiple conducting paths. Additionally in low strain levels such the presented ones (0 to 0.8%), the resistance change is mainly caused by the geometric deformation of the specimen rather than the damage accumulation.

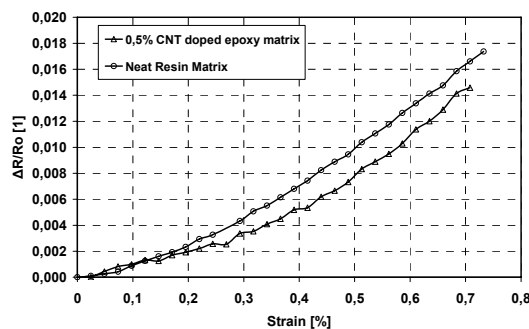


Fig. 1 Normalized Resistance change ($\Delta R/R_0$) vs. strain of quasi-isotropic CFRP laminates with neat (left) and 0,5 % CNT doped (right) epoxy matrix

The ability of the electrical resistance monitoring to follow directly the applied strain is verified also during the tension-tension fatigue tests. The adequate sampling rate of the logging system of the multimeter used revealed the aforementioned ability as presented on right part of Fig. 2.

Additionally the continuous recording of the resistance during the complete time of the fatigue tests up to the final specimen rapture as presented on left part of Fig. 2 bring in front very interesting results about the correlation of the fatigue damage with the specimens resistance. It can be stated that the mean –strain free- resistance is affected by the induced fatigue cycles. During initial stages of loading it can be observed that resistance is dropping, stating an unexpected increase of material’s conductivity, afterwards it follows a positive slope up to final fracture as expected from the material damage built-up.

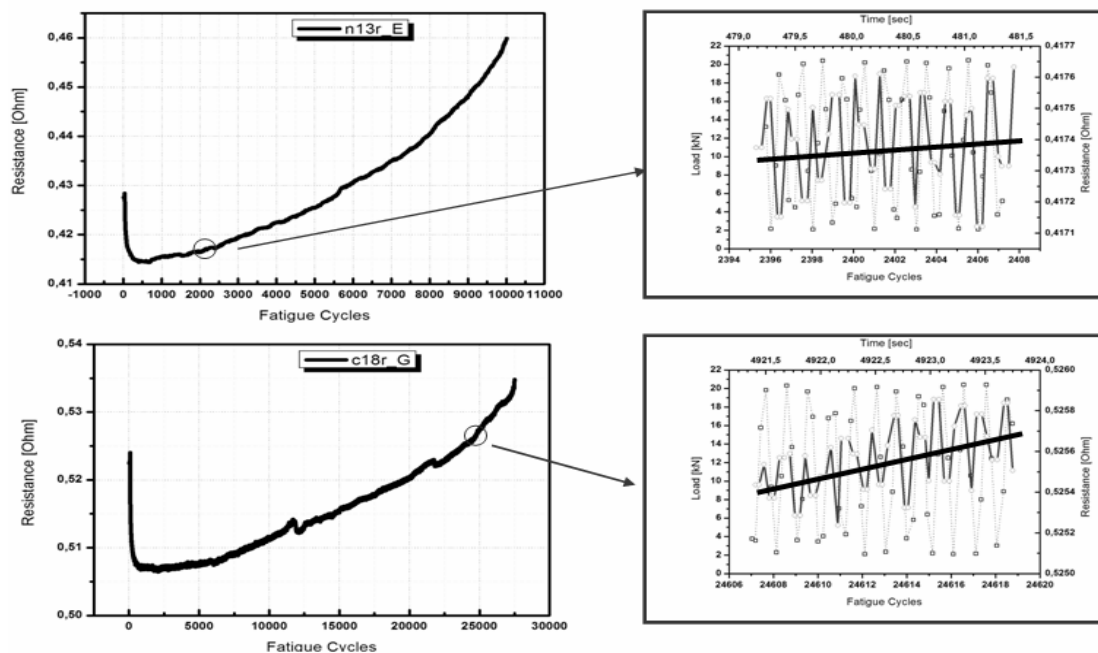


Fig. 2 (left) Resistance Change versus fatigue cycles. (right) Combined graphs of applied fatigue load and resistance versus experimental time and fatigue cycles.

The sudden drop of the resistance during initial stages -as also clearer showed in Fig. 3 (left) - created strong indications for two counteractive mechanisms during this stage. The first mechanism is the damage accumulation that is expected even in the initial stages and the second mechanism is the self-alignment of the conducting network of the material due to new electrical fiber contacts created after initial interlaminar matrix cracking.

In Fig. 3 (right) the electromechanical behavior of both types (neat and doped epoxy matrix) is summarized for comparison. It seems that neat epoxy specimen has a wider range of resistance change before fracture than the doped specimen. This can be explained by the presence of the electrical conductive CNTs doped inside the matrix, that keep doped composite’s conductivity at higher levels than the undoped one during all fatigue test. Additionally the smooth and quite linear behavior of resistance after the first stage gives a strong point for the use of the method for the material damage monitoring.

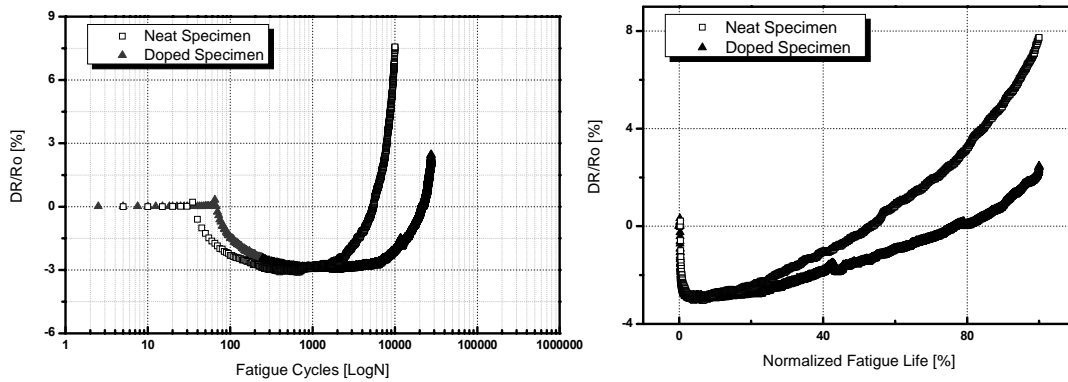


Fig. 3 (left): Normalized Resistance Change versus fatigue cycles in logarithmic scale (right) : Normalized Resistance Change ($\Delta R/R_o$) versus the normalized fatigue life

The promising results for the electromechanical behaviour are accompanied with interesting results for enhance mechanical performance as received from standard tensile and tension-tension fatigue tests. According to the performed tensile tests (ASTM D-3039) in Fig. 4, there is an 11% increase in the Young Modulus of the CFRP with the doped epoxy matrix from the reference. On the contrary, the authors observed that the maximum stress was kept at the same levels with the doped CFRP exhibiting a slight reduced mean value.

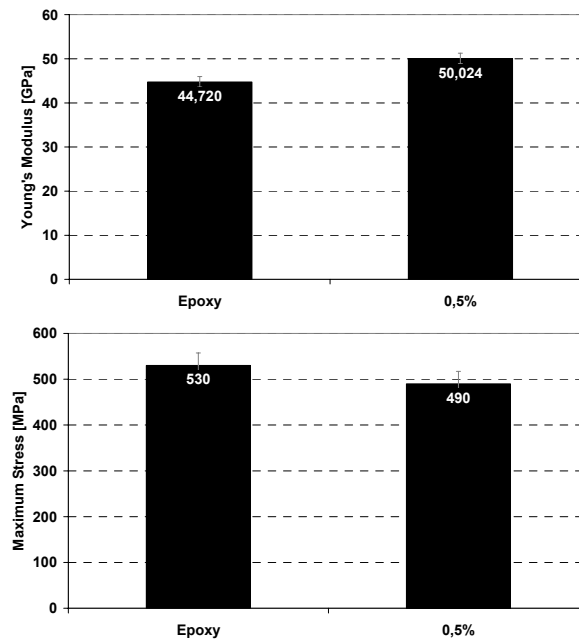


Fig. 4 (left): Experimental Young Modulus (right) Maximum Stress at break , observed during tensile tests for quasi-isotropic CFRP with neat and 0,5% doped epoxy matrix

Additionally the tension-tension fatigue tests conducted according to ASTM D-3479, indicated that doped matrix composites exhibit an increased fatigue life for same stress levels as seen in Fig. 5 (up). Moreover the right part of Fig. 5, using as infinitive fatigue life the 10^6 cycles and forecasting the fatigue limit using a logarithmic fitting curve, it is demonstrated that the doped quasi-isotropic CFRP had a increased fatigue limit (72%) compared to the reference CFRP that had 64%.

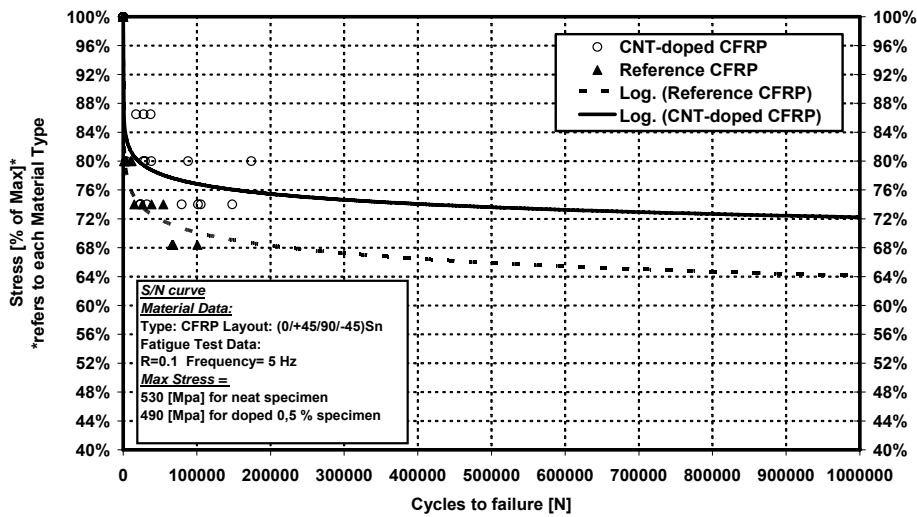
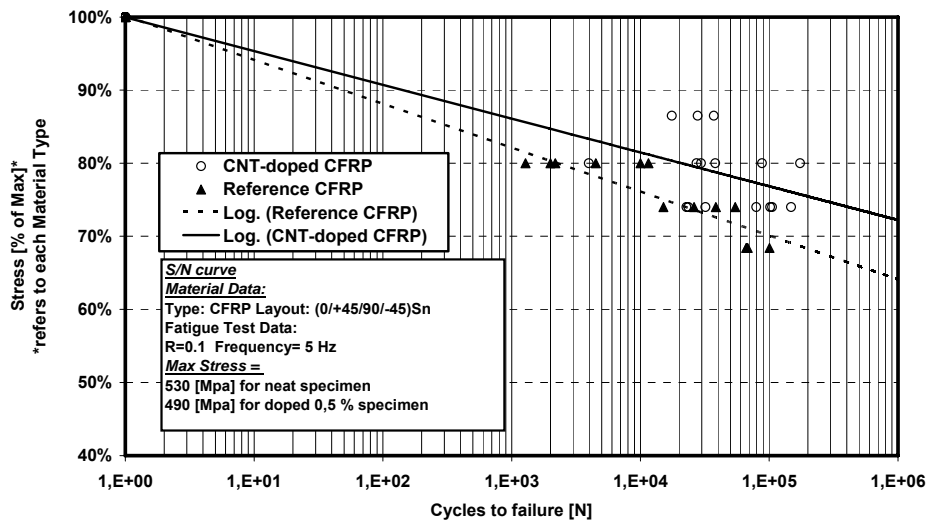


Fig. 5 Fatigue S-N curves, Stress (S) shown as percentage of the maximum static tensile stress and fatigue cycles (N) expressed in logarithmic (up) and linear (below) form.

As about the parallel AE monitoring, the AE activity of two typical specimens (CNT doped and neat) is summarized in figures 6 and 7. Four graphs are drawn for each specimen: a) Amplitude versus test time, b) Amplitude distribution over test time, c) Cumulative number of AE hits versus test time, d) Cumulative AE energy versus test time

For the neat specimen, the AE activity is kept at high levels during the first 1100 sec (5500 cycles) as figure 6b shows. After this point the AE activity lowers. In the case of the doped specimen, the AE activity rises gradually during the first 1100 seconds and then it seems to be kept constant up to failure.

In both material systems the temporal moment around 1100 sec (5500 cycles) seems important for the damage accumulation in the material and may be correlated with the matrix cracking saturation.

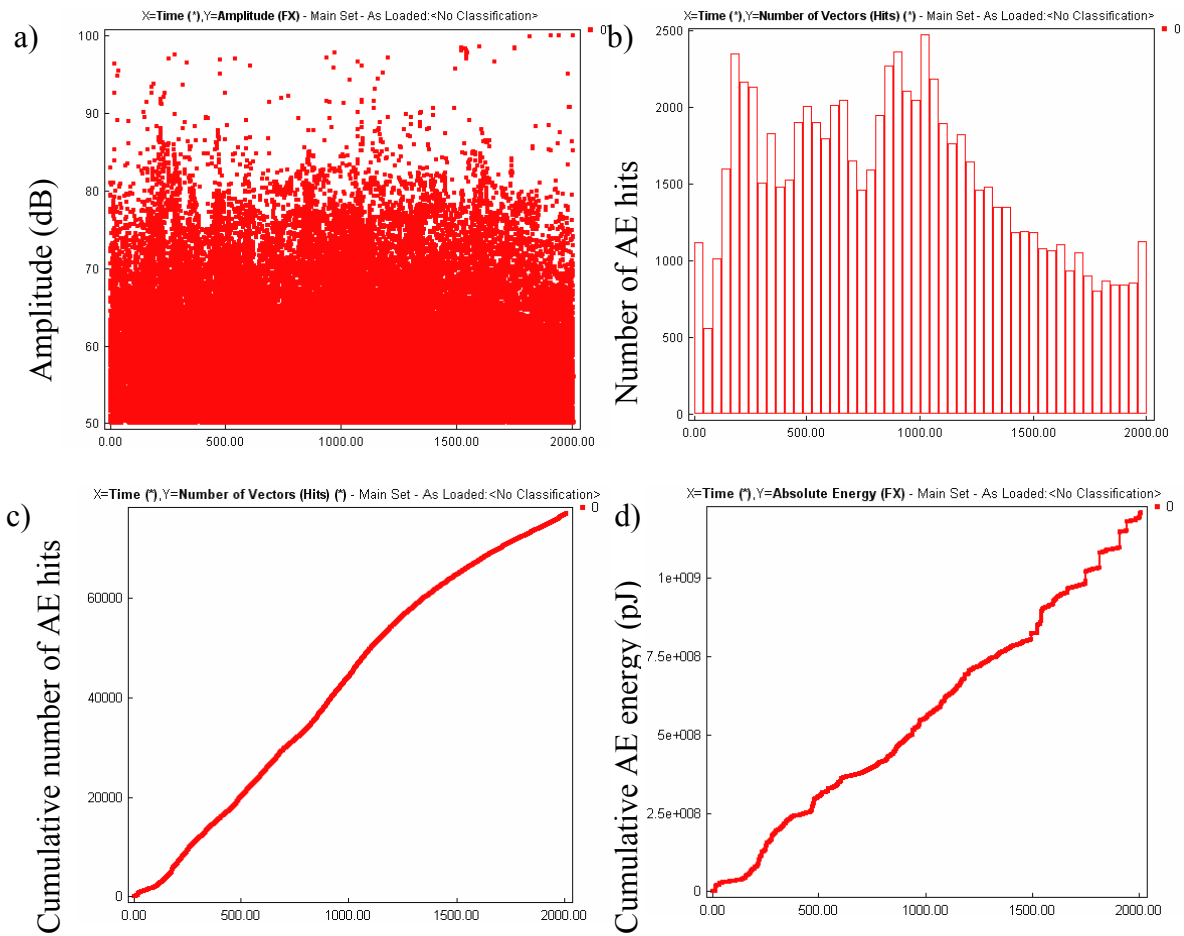
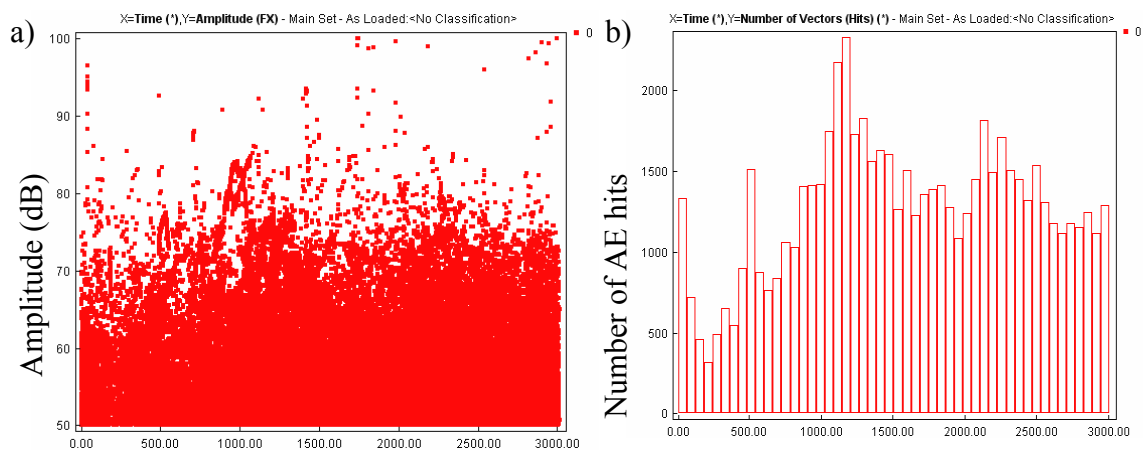


Fig. 6 For Neat Specimen, a) Amplitude versus test time b) Amplitude distribution over test time c) Cumulative number of AE hits versus test time d) Cumulative AE energy versus test time [in all graphs x axis is time in seconds]



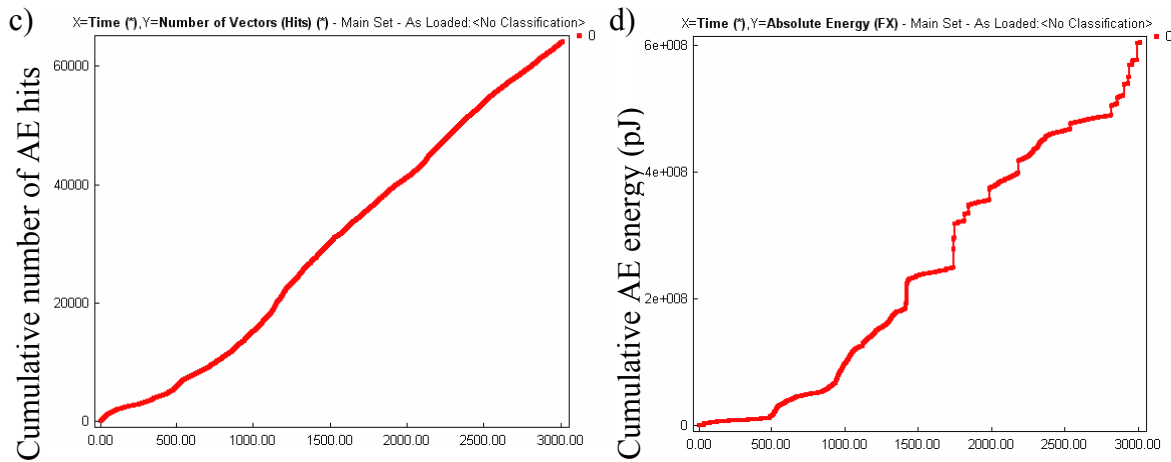


Fig. 7 For Doped Specimen, a) Amplitude versus test time b) Amplitude distribution over test time c) Cumulative number of AE hits versus test time d) Cumulative AE energy versus test time [in all graphs x axis is time in seconds]

4. CONCLUSIONS

The real time sensing of load variations via electrical resistance measurements is verified for quasi-isotropic composites in both cases, with 0.5% doped epoxy matrix and with reference matrix. It is also confirmed that the mean resistance changes during the tension-tension fatigue test could reflect the damage accumulation of both materials. Two main stages are distinguished. During the initial stages of the fatigue, less than 10% of the fatigue life, the resistance is suddenly dropping mainly due to the self-aligning of the conducting network of the material with new electrical fiber contacts created after initial interlaminar matrix cracking. Having the conducting network reached a new equilibrium; resistance is mainly affected by the more intense damage accumulation and is increased continuously up to the final breakage. The effect of the temperature profile during fatigue could also affect the resistance change, but it can be neglected as minor for this case since for quasi-isotropic composites the carbon phase is dominant in all conduction mechanisms and the temperature coefficient is approximating the small values of carbon itself.

As about the mechanical performance, the doped epoxy matrix composites exhibited an 11% increase in the Young Modulus of the CFRP with the doped epoxy matrix from the reference. On the other hand, the observed maximum stress was at the same level with the doped CFRP exhibiting a slight reduced mean value. A very promising fact is that the fatigue life for same stress levels is also increased for the doped composites with a corresponding augmentation of the fatigue limit.

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