



Non-intrusive health monitoring of infused composites with embedded carbon quantum piezo-resistive sensors



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ABSTRACT

Fibre reinforced polymer composites (FRP) are developing faster than ever in fields such as aeronautics, automotive, naval or energies requiring materials with high toughness/weight ratio, to lower their environmental footprint. However, one brake to FRP development is that their damage can initiate without warning signs, due to unexpected overload, chocks, fatigue or defects. Structural health monitoring (SHM) aims to secure their use by anticipating their catastrophic fracture and make predictive maintenance. Existing solutions are using combinations of fibre Bragg gratings and ultrasonic sensors to follow strain evolution and cracks propagation in the composite.

Here we present a new generation of nanocomposite quantum resistive sensors (QRS) that can be homogeneously introduced in FRP to sense their health without sacrificing their integrity. QRS as thin as 1.5 μm , have been implemented on E-glass fibres (GF) before infusion of epoxy resin (EP). They allow to follow both strain and damages in both elastic and plastic domains up to failure with high sensitivity, i.e., gauge factors (GF) up to 6.

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1. Introduction

Fibre reinforced polymer composites (FRP) are developing faster than ever particularly in fields pushing to reduce costs and/or consumption and increase the performance/weight ratio. The green energy industry has designed giant offshore turbines with resp. 73.5/80 m blades, rotor diameter of 150/164 m and power of 6/8 MW [1, 2]; the aeronautics has developed planes with 50% in weight of composite [3, 4], and even larger composite fraction in helicopters [5] or solar powered planes [6]. More recently, the automotive industry with the development of hybrid [7] or electrical vehicles [8], and the building area with the rehabilitation of concrete structures [9] have also followed the same evolution. However, due to their anisotropic structure, FRP fail in a complex manner making their maintenance, non-destructive evaluation, i.e., structural health monitoring (SHM) still highly challenging. Thus, there is a strong need for an efficient and reliable SHM method capable to increase their reliability and lifetime.

Lots of efforts have been made over the last decades, regarding early monitoring of micro-crack propagation in the materials.

Traditional sensors, like commercial strain gauges, can measure the strain only in defined directions and locations at the surface of the composites [10]. These commercial strain gauges remain expensive and commonly exhibit a gauge factor around 2; moreover they suffer from a tendency to be detached from their substrate during use [11]. Thus, they are unable to predict any internal damage of the composites or cracks initiation and damage accumulation, which limits their application. Therefore, to obtain an intricate picture of the entire structures' health, sensors organized in network should be ideally placed at different locations on the surface and in the core of the engineering structure, after the points of maximum solicitations have been determined by modelling. Additionally an optimal sensor network should be robust, provide real-time, accurate information on the structure health to the operator, but in a non-intrusive way not to weaken the composite by compromising its integrity.

Early works of self-sensing of advanced composites made by Chung et al. [12, 13], and Schulte [14, 15], in the beginning of last decade, were limited to only electrically conductive carbon fibre reinforced polymer composites. But matrix-dominated fracture mechanisms can hardly be monitored utilizing such conductive fibres and hence restrain their applications. Since then, health monitoring in electrically non-conductive polymer composites

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have been reported using embedded fibre Bragg grating sensors (FBG) [16] [17, 18]. Fibre Bragg gratings can measure very precisely localized strain fields, thanks to changes in the strain-optic-coefficient of refraction when mechanically deformed. This leads to a characteristic shift in Bragg-wavelength that can be detected by transmission of light, which passes through the fibre. This sophisticated method can be used for in-situ real time measurements, but requires expensive instrumentation for the analysis of the signals. Moreover, due to their sensitivity to moisture and chemicals they need to be protected by a polymer sheath that will finally make their diameter become at least ten times larger than that of reinforcement fibre. It is thus not exclude that they could introduce localised weakness points in the composite part. Additionally, the production costs associated to the mapping of large parts with such kind of optical fibre network may not be acceptable. Finally, if damages or cracks happen in different locations without crossing one of the FBG sensors, it cannot be detected [19, 20].

The discovery of carbon nanotubes (CNT) by Radushkevich et al. in 1952 [21], and their further development by Iijima in 1991 [22], have opened a new era of nanotechnology. Due to their unique physico-chemical properties, very high electrical and thermal conductivity [23, 24], exceptional mechanical properties [25–27], high aspect ratio [28], CNT have brought about new prospects for the design of smart materials [29]. Particularly, their dispersion into polymer matrices to make conductive nanocomposites allowed to reach innovative applications such gas barrier coatings [30], vapour or strain sensors able to monitor respectively humans [31] or composite's [32, 33] health to anticipate their damage.

In case of glass fibre reinforced polymer composites (GFRP), all components being electrically insulating, it is possible to structure a conductive network by percolating carbon nanotubes (CNT) in the polymer matrix [34] or in fibres' sizing [35], that can be used to sense cracks initiation and propagation. Thostenson & Chou have been the pioneers of the development of conductive networks in GFRP using embedded carbon nanotubes together with the modelling of their damage sensing [36–40]. In this context, Gao et al. [41] coupled piezo-resistive and acoustic emission characterization methods to monitor the onset and propagation of damage in composite materials. Nevertheless, the difficulty to obtain a homogeneous dispersion of CNT in the matrix and the strong increase of viscosity resulting from their introduction at contents superior to 1.5 wt% make the fabrication of such materials difficult using existing manufacturing tools such as infusion for example. Moreover, it remains difficult to identify the location of crack initiation and propagation as the nanotube sensor network provides only information of the overall structure. Other alternative approaches like the incorporation of CNT random networks known as buckypapers [42, 43], CNT grown on Al_2O_3 $\mu\text{spheres}$ [44], CNT/polymer films [45], CNT/polymer fibres [46] and CNT coated polymer fibres [47, 48], conventional layer-by-layer (LbL) process for multilayer thin film generation with several polyelectrolytes have already been reported [49, 50], for measuring strain and damage in advanced composites. Due to several shortcomings of CNT film sensors or CNT/polymer film sensors like fragility, weak bonding between the CNT and polymer via van der Waals attraction, improper adhesion between sensor and host substrate [51], restrain their uses in real structural applications. It is also possible to use a multilayer sensitive thin film assembled layer-by-layer (LbL) by successive dipping in polycations and polyanions solutions, but this fabrication process is more complex and makes the control of the initial resistance less easy. Atomic Force Microscopy (AFM) [52] and Raman spectroscopy [53, 54], were also reported to have been for health monitoring of structural composites, but are they are impractical in the real field of application due to the difficult implementation of the measuring equipment and its high cost.

Other solutions used in epoxy based composites result in the development of a CNT rich conductive interphase directly on the surface of single fibres by dip coating or electrophoretic deposition (EPD) for glass fibres [55, 56], or by in-situ CVD growth for carbon fibres [57]. Nevertheless, the control of architecture, thickness, initial resistance of the sensor remains quite difficult. This overview shows that despite the huge potential of nanocomposite sensing, it is still challenging to fabricate intelligent self-sensing composite materials for detecting localised stress/strain concentration, without any detrimental effects on the integrity of the structure, the process or cost of structural composites.

In this paper the fabrication of intelligent self-sensing composites with embedded sensor has been reported. The formation of conducting polymer nanocomposite (CPC) piezo-resistive sensors at the fibre/matrix interface using sLbL hierarchical assembly with large, stable and reliable electrical responses has been described. This technique already demonstrated many advantages in the realization of nanocomposites sensors [58–65]. Firstly, this solution process allows to well disperse CNT in many solvents and polymer matrices (no need of polyelectrolytes), then it makes possible to build in situ sensing skins on almost all kinds of surfaces, rough, flexible, large, in different locations needing monitoring (with good adhesion); finally the step-by-step control of the formation of a hierarchically conducting architecture gives several adjustment parameters, useful to tailor piezo-resistive properties.

In this context, the present study reports on the implementation of CPC transducers located in the core of composite samples to provide them with self-sensing capability. This strategy of health monitoring tested on glass fibres/epoxy composites is quasi non-invasive and compatible with infusion process. The strength of CPC sensors technology is to allow the following of composites health in both the elastic and plastic domains up to ultimate failure, without compromising their integrity. Their fabrication by spray layer by layer is convenient as it allows a good control of sensors electrical properties and a targeted deposition directly onto glass fibres in areas of interest for damage location.

2. Experimental details

2.1. Materials

Multiwall carbon nanotubes (NC-7000 MWCNT) were kindly provided by Nanocyl (Belgium). This grade corresponds to MWCNT with an average diameter of 10 nm and a mean length comprised between 100 and 1000 nm. Epolam 2020 epoxy resin & amine hardener were purchased from Axson, France. Chloroform (99%) was purchased from Aldrich (France) and Taffetas E-glass fibre ($0^\circ/90^\circ$, 165 $\text{gm}\cdot\text{m}^{-2}$) from Gazechim France.

2.2. Techniques

The sensor's thickness was measured by Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM). AFM was performed at ambient conditions using light tapping mode (TM-AFM) on a di-calibre multimode scanning probe microscope from Bruker-Veeco. The set point amplitude of antimony-doped silicon tapping mode cantilever (LTESP model, Veeco, USA) was about 4.5 V. The cantilever with tip radius between 5 and 20 nm had typical resonance frequency about 270 kHz, and a cantilever spring constant (k) was 20–80 $\text{N}\cdot\text{m}^{-1}$. Scanning Electron Microscope was a Jeol series (JSM-6031F) model.

A servo-hydraulic Instron 5566A was used to perform static tensile experiments. Deformations were measured using a 10 mm extensometer, at a crosshead speed of 0.2 mm/s.

Dynamic tensile testing was performed with Instron 8800

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