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Graphene-functionalized Polymer Composites for Self-sensing of Ultrasonic Waves: *An Initiative towards "Sensor-free" Structural Health Monitoring*

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Abstract

With recognized bottlenecks of guided ultrasonic wave (GUW)-based structural health monitoring (SHM) for composites, conventional polymers are nano-engineered and endowed with capability of self-perceiving GUWs. A built-in sensing network is formed with graphene nanoparticles, optimized and diffused in fibre-reinforced polymers, in which the quantum tunneling effect can be locally triggered when GUWs traverse the composites. The diffuse sensing network makes it possible to acquire GUWs at any site of the functionalized composites, avoiding use of conventional ultrasonic transducers that must be externally attached to or internally embedded in the composites. With an optimized nano-structure, the functionalized composites have been demonstrated self-responsive to GUWs up to 500 kHz. In experimental validation, GUWs propagating in a glass fibre/epoxy laminate are self-sensed by the laminate at the sites arbitrarily selected, to observe no discrepancy against counterpart signals obtained with piezoelectric sensors. To take a step further, barely visible impact damage (BVID) in the laminate is located accurately using the self-sensed GUW signals. This study has spotlighted a new breed of functional polymers with capability of self-health monitoring, without using external sensors. The use of associated cables and wires is also minimized. Not only does it facilitate a reduced weight/volume penalty to the original composites, but also minimizes mechanical degradation of the composites due to the intrusion of sensors, blazing a potential trail in developing "sensor-free" SHM for composites.

***Keywords*:** functional composites; graphene nanoparticle; self-sensing; guided ultrasonic waves; structural health monitoring

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1. Introduction

The biological nervous network – the most complex yet mysterious system known in the universe – is a supreme model to follow when scientists and engineers contrive, design, and optimize sensing systems in engineering practice. A neural network is an intricate and sophisticated collection of diffused nerves and neurons, activation of which defines a recognizable linear pathway carrying messages to and from the brain/spinal cord to body parts. In virtue of the nervous systems throughout the body, a biosome can, by nature, precisely perceive ambient fluctuation and sense self-body status, on which basis the cognition and judgement can be subsequently made.

In pursuit of farthest simulation of a biological nervous system, continued effort has long been made for diverse applications in which the sensing plays a crucial role, and there is no exception for structural health monitoring (SHM) – an emerging, bionic technology with an initiative to improve reliability and safety of engineering systems, accompanying significant reduction in life-cycle costs. Effective SHM targets prospective life extension of an ageing structure via real-time, continuous, *in-situ* monitoring of its integrity. In SHM an elaborately designed sensor network, in either a sparse or a dense fashion, is to be externally attached to or internally embedded in the structure under inspection, to acquire desired information. To configure a sensor network, commercially available sensors are legion, as typified by lead zirconate titanate (PZT) wafers, optical fibres, strain gauges, electromagnetic acoustic transducers, and polyvinylidene fluoride (PVDF) piezoelectric films to name a few.

However, with inevitable intrusion to the original structure, the sensors *per se*, in whichever type, may degrade the structural integrity to some extent, regardless of their intended role of detecting defect- or damage-caused structural deterioration. A multitude of sensors are often

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networked to form a dense sensor network with a hope of acquiring rich information, but integration of a dense sensor network unavoidably commits severer intrusion to the structure. This possibly lowers local material strength, introduces defect/stress concentration/debonding, downgrades resistance to corrosion, and imposes weight and penalty to original structures due to unwieldy cables and wires used in a sensor network (for correlating individual sensors). Even worse, such a kind of degradation due to sensor intrusion is progressive and intensified when structures are operated in a cruel service environment. Under cyclic loads, an intruded sensor network runs an extremely high risk of de-attaching from the host structure, owing to degradation of the adhesive layers between the host structure and sensors. To minimize such a risk, a limited number of sensors are placed at strategic sites to form a sparse sensor network, assisted with specific signal processing algorithms to manipulate "limited" information acquired. Inferior to a dense network, a sparse sensor network is understandably incapable of offering high-precision monitoring.

The recent advances and technological breakthroughs in emerging nanotechnology have ushered a promising avenue to develop new breeds of sensors capable of accommodating demanding sensing requirements for high-precision SHM [1-4]. Prominently lightweight and small, nanocomposites-inspired sensors degrade structural integrity at a minute degree, whereas offering additional merits including low cost and ease in fabrication. The nanocomposite sensors have found their superb niche in measurement, detection and monitoring, and favorable examples include measurement of material strain under quasi-static loads [4, 5] or under low-frequency dynamic loads [6, 7], detection of gas content leakage [8-10], and identification of structural damage [11-13]. In these paradigms, nanocomposite sensors have demonstrated their higher sensitivity and precision than

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conventional metal strain gauges or gas sensors. Representative, Zeng *et al.* [14] developed a carbon black/polyvinylidene fluoride sensor that can perceive dynamic strain in a broadband frequency regime from static strain, through medium-frequency structural vibration, to high-frequency ultrasound. Liu *et al.* [15] attempted a new nanocomposite sensor, coatable on a structural surface, to replace conventional PZT wafers, and the sensor shows fidelity, ultrafast response and high sensitivity to acoustic-ultrasonic signals. The captured signals are comparable with those obtained with commercial PZT wafers. The coated sensors can further be networked for implementing acousto-ultrasonic-wave-based SHM. Though holding vast potential to strike a balance between the sufficiency of information (as much as possible) and the usage of external sensors (as few as possible), signal acquisition using thus produced sensor networks is still based on discrete measurement with individual sensors. Moreover, the reliance on cables and wires to network individual sensors is still persistent. The use of printed circuits [16, 17] may, to some extent, reduce the weight and volume of cables and wires, but the detachment of circuits from the host structures under cyclic loads remains another predicament.

Inspired by natural biological nervous systems, conventional composites have been tailor-functionalized, in which the reinforcements, such as carbon fibres or conductive particles dispersed in matrix, are exploited to sense loads applied to the composites, endowing the composites with a sort of capability of "self-sensing". "Self-sensing" in this study emphasizes an intrinsic capability of the functionalized polymer composites for sensing changes in material conditions (*e.g.* strain, stress, damage, temperature, *etc.*) in terms of the measured changes of particular properties of the material itself such as piezoresistivity [18, 19]. Exemplarily, Kupke *et al.* [19] used continuous carbon fibres in polymer as sensing elements to measure changes in electrical resistance caused by external loads or fibre

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breakage. It is noteworthy that using carbon fibres themselves as sensing elements is only effective to evaluate severe damage and in particular the fibre breakage, whereas uncapable of detecting the onset of matrix damage, matrix cracking or delamination. Ruschau *et al.* [20] extended the same sensing philosophy to glass fibre-reinforced composites, in which conductive particles were dispersed into the matrix to form a conductive network for sensing change in strain. This study accentuated that a sensitive sensing network could be formed only when the particles are sufficient, implying a great percolation threshold is expected. For example, the percolation threshold is 50 wt.% if Ag particles are opted for to form a sensing network [21]. A great threshold (namely the use of a large quantity of conductive particles) brings drastic weight increase to the original composites. To minimize weight penalty from added conductive particles, lightweight nanoparticles such as carbon nanotubes, carbon black, and graphene, have gained prominence [11, 13, 22, 23], in which the percolation thresholds for most nanoparticle-formed conductive networks are less than 10 wt.%. Thanks to the recent advances in nanomaterials, the "self-sensing" capability has been extended to fibre-reinforced polymer composites through dispersing conductive nanoparticles in polymer matrix. The measured piezoresistivity can then be used to calibrate changes in strain and stress or indicate the occurrence of material damage [24-26].

Nevertheless, none of the existing self-sensing networks in polymer composites, using either continuous fibres or conductive particles, is responsive to an applied load of a frequency beyond kilohertz, let alone ultrasound signals of which the frequencies are usually of several hundred kilohertz or even megahertz, along with an ultralow signal magnitude (in the order of microstrain). This deficiency has considerably restricted the "self-sensing"-driven SHM in a low-frequency regime (*e.g.*, static electrical resistance tomography [27-29], quasi-static loading-based detection [13] and low-frequency vibration-based monitoring [30]). It is

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envisaged that the methods operated in a low-frequency range are usually unwieldy to identify embryonic damage of small dimensions such as the onset of delamination.

In recognition of the bottlenecks of today's "self-sensing" composites, sensing networks are formed using two-dimensional (2-D) graphene nanoparticles, and diffused in conventional fibre-reinforced polymer composites, by extending the authors' earlier work on nano-engineered sensors for ultrasonics-based SHM [14, 15, 31]. In virtue of the quantum tunneling effect in the sensing network triggered by a traversing ultrasonic wave, the functionalized composites are endowed with capability of self-perceiving guided ultrasonic waves (GUWs) at any interested site of the composites, avoiding the use of bulky ultrasonic sensors that are externally attached to or internally embedded in the composites. Experimental validation is conducted, in which GUW propagating in a glass fibre/epoxy laminate are self-sensed by the laminate at the sites arbitrarily selected. Propagating GUWs carry rich information pertaining to the health status of the composites, extraction of which can facilitate quantitative evaluation of damage in the laminate. Addressing a new concept of "diffuse sensing", this approach attempts an initiative of "sensor-free" SHM.

2. "Self-sensing" Composites: Principle and Fabrication

2.1 Mechanism of "Self-sensing"

Upon evenly dispersed in a dielectric polymer, conductive nanofillers, such as 2-D graphene nanoparticles, can create an electrical network in the polymer. The electrical properties of the composites can be described, according to the percolation theory [32], as

$$\sigma \propto \left(p - p_c \right)^t, \tag{1}$$

where σ signifies the electrical conductivity of the nanofiller-dispersed composites, p the

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volume fraction of nanofiller, p_c the percolation threshold of the composites, and exponential t a constant associated with the composites. The percolation threshold represents a critical volume fraction of the nanofillers, beyond which a slight increase in the nanofiller content can give rise to a tremendous leap in the conductivity of the composites [32]. In a nanoparticle-formed conductive network, the electrical resistance consists of three key components: the intrinsic resistance of nanoparticles ($R_{nanoparticle}$), the constriction resistance owing to direct contact of nanoparticles ($R_{contact}$), and the tunneling resistance between two neighbouring nanoparticles (R_{tunnel}), namely [20]

$$R = R_{nanoparticle} + R_{contact} + R_{tunnel}\,. \tag{2}$$

In particular, the tunneling effect – a quantum mechanical effect – describes the phenomenon that when the insulative barrier between two neighbouring conductive nanoparticles becomes thinner than a critical threshold, a tunneling current can be triggered when electrons move through a barrier that they classically should not be able to move through [33-35]. It is the quantum tunneling effect that brings somewhat appealing and unique properties to nanocomposites such as semi-conductive properties. In Eq. (2), R_{tunnel}, is defined by [36]

$$R_{tunnel} = \frac{V}{AJ} = \frac{2h^2 d}{3Ae^2\sqrt{2m\lambda}} \exp\left(\frac{4\pi d}{h}\sqrt{2m\lambda}\right), \tag{3}$$

where V denotes the electrical potential difference, J the tunneling current density, A the cross-sectional area of the tunnel, h the Planck's constant, d the distance between two nanoparticles, e the quantum of electricity, m the mass of electron, and λ the potential height of insulating layer (0.5-2.5 eV for epoxy in this study). The tunneling resistance, R_{tunnel}, depends on the thickness and material properties of the insulating layer, which is much higher than the other two types of resistance ($R_{nanoparticle}$ and $R_{contact}$) by a multitude of orders of magnitude. The tunneling effect is particularly prominent when two nanoparticles are in a close proximity (of the order of several nanometers) but not in a direct contact. This stresses

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that at the percolation threshold, the quantum tunneling effect dominates the electrical resistance manifested by the nanocomposites [1, 37-39]; beyond the threshold, nanoparticles become contacted or overlapped, under which the tunneling effect tends to be weak and the conductivity of the nanocomposites saturates.

GUWs, high-frequency elastic waves guided by thin plate-like waveguides, feature high sensitivity to structural damage, omnidirectional dissemination, fast propagation, and strong penetration through waveguide thickness. In virtue of these features, GUWs have identified their superb applications in high-precision SHM. As GUWs traverse a nanocomposite waveguide, the wave-induced strain modulates the inter-distance among nanoparticles and consequently triggers the quantum tunneling effect if the nanoparticle-formed conductive network is close to its percolation threshold, which alternates the measured piezoresistivity.

It is such an electro-mechanical property of the nanocomposites that serves the underlying mechanism to be harnessed in this study, on which basis the conventional composites are functionalized, and endowed with a capability to respond to GUWs, as illustrated schematically in **Figure 1**. It is noteworthy that the GUW-induced strain features an extremely low magnitude that is of the order of several micro-strain, under which the tunneling effect is the key mechanism resulting in change in the conductivity of the nanoparticle-formed sensing network as interpreted earlier. In virtue of the GUW-triggered tunneling effect in the nanoparticle-formed percolating network, properly fabricated and optimized nanoparticle-dispersed composites can warrant desired sensitivity to propagating GUWs.

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2.2 Development and Optimization of Self-sensing Network

Driven by the above-mentioned sensing philosophy, 2-D graphene nanoparticles are selected as the nanofiller to develop the "self-sensing" network, on the ground of a twofold fact, when compared against other candidate nanofillers such as carbon nanotube or carbon black,

(i) less entanglement and aggregation: the 2-D morphology of graphene nanoparticles facilitates significant reduction in particle entanglement and aggregation, as can be observed in the scanning electron microscope (SEM) image shown in **Figure 2a**; compared with rope-like nanofiller types such as carbon nanotubes, **Figure 2b**, the less entanglement and aggregation can be conducive to creating an even, stable and uniform percolating network in the matrix materials. Thus, though some theory studies have stated that rope-like nanoparticles usually percolate at lower filler content than disk-like nanoparticles do [40], real composite morphology is often found contrary [41]. Moreover the processes required to disentangle nanotubes are not cost-effective, which may shorten the original nanotubes and further impede the formation of a percolating network [42];

(ii) a greater surficial contacting area and a higher lateral-thickness aspect ratio: compared with sphere-like nanofiller types such as carbon black, the 2-D nanoparticles give rise to a lower percolation threshold [43] – that is because the conductive network formed by sphere-like nanoparticles is based on fractal aggregates as seen in **Figure 2c**; moreover the lower surficial area of sphere-like nanoparticles makes it difficult to engender sufficient conductive paths in a sensing network. Similar results have also been reported in many places and connected to the electrical properties [44-46].

2-D few-layer graphene nanoparticles with a thickness of ~3 nm and a lateral size of ~10-50 μm are obtained from TANFENG Graphene Tech Co., Ltd., China. Epoxy resin (Araldite®

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GY 251) and hardener (Aradur® HY 956) are mixed at a mix ratio of 5:1 by weight to develop the matrix. The epoxy resin is first placed on a hot plate and heated up to 80°C to decrease its viscosity. 2-D graphene nanoparticles are uniformly dispersed in the matrix to form an even conductive network using a direct mixing method. Mechanical stirring is applied to mix graphene nanoparticles into the matrix for 30 minutes, followed with a sonication in a bath-type sonicator for another 5 minutes. Consequently, plain-woven glass fibre fabrics (Colan AF 218) are impregnated with the graphene-compounded epoxy using a hand roller by a hand lay-up process, in **Figure 3**, to produce a series of identical 8-layer laminates with a thickness of ~1.4 mm each. Upon curing at a room temperature for 24 hours following a standard vacuum bagging procedure, the graphene-diffuse glass fibre-reinforced composite laminates are readily available.

In the above a direct mixing method is used to mingle graphene nanoparticles with matrix. In earlier work [47-49], solution-based methods are often employed to disperse nanoparticles in a polymer, in which a large volume of solvents (acetone, chloroform, N-methyl-pyrrolidone, dimethylformamide, *etc.*), which is usually 10-1000 times the volume of that of the polymer itself, has to be consumed, in order to break up nanoparticle agglomeration and entanglement. Good for a small amount of production, these methods, however, are generally infeasible to manufacture laminated nanocomposite structures because such a process entails a large amount of nanoparticle-filled epoxy resin, incurring excessive cost and being environmentally unfriendly. To circumvent this, a direct mixing method is adopted to disperse graphene nanoparticles into polymers, warranting even and uniform dispersion without obvious nanoparticle agglomeration (note that it is however not the case for rope- or sphere-like nanoparticles (e.g., carbon nanotubes or carbon blacks), in which a direct mixing method may incur prominent particle entanglement and bundling).

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GUWs are of high frequencies yet ultralow magnitudes, and GUW-induced strains are usually insufficient to trigger the quantum tunneling effect in a graphene nanoparticles-formed conductive network without appropriate structural optimization. To develop an optimal conductive network that is most sensitive to GUWs, the above graphene-diffuse glass fibre-reinforced composites are comparatively fabricated with different percentages of graphene, whereby to ascertain the percolation threshold at which the tunneling resistance, R_{tunnel}, dominates, as interpreted in Section 2.1. This is different from those cases when relatively large strain, such as that induced by structural vibration, is to be measured, in which the change in electrical resistance is mainly attributable to the disconnection among nanoparticles and breakage of electrical paths in the conductive network (namely, the change in $R_{contact}$).

At different percentages of graphene nanoparticles ranging from 0 to 5 wt. % (seen in **Table 1**), the conductivity (σ) of the fabricated composites is measured using a standard two-point volt-amperometric method with a digital multimeter (Keithley® DMM 7510). Silver conductive paints (SPI® Z05001) are pre-coated on the fabricated samples to create electrodes. Here σ is calculated according to

$$\sigma = \frac{L}{R\xi}, \tag{4}$$

where R signifies the measured resistance (as defined by Eq. (2)), ξ the cross-sectional area, and L the distance between two electrodes. Four identical samples at each content are tested, yielding a correlation as plotted in **Figure 4**. A marked increase, by a six-order of magnitude, in the conductivity of the composites can be observed when the content of graphene nanoparticles is between 0.5 and 1 wt%, and this indicates substantial formation of a

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percolating network within the composites. According to the percolation theory [43], the percolation threshold can be determined as 0.8647 from the power-law function curve fitting based on Eq. (1). The formation of a percolating network warrants the response sensitivity of the sensing network to GUWs. The percolation region is generally linked to the highest sensitivity of nano-engineered composites [5, 50]. Specifically, for sensing GUWs, the amplitude of a captured GUW signal has been demonstrated to reach its maximum when the graphene nanoparticle content is at the percolation threshold, as reported in authors' future work.

3. "Sensor-free"-inspired SHM

Conventionally, GUWs-based SHM employs miniaturized sensors such as PZT wafers or optical fibres that are internally embedded or surface-mounted on an inspected structure, serving as GUW actuators or receivers. These sensors are collocated into a transducer network via cables and wires [51, 52]. Considering practicability, actuators and sensors have to be prudently positioned at strategic sites of an inspected structure.

Distinct from this conventional means of configuring a sensor network for GUWs-based SHM, the developed composites with self-sensing capability substantially eliminate such a need to install external sensors for GUW acquisition. Notably, GUW signals can be acquired any site of the composites as the nanoparticle-formed sensing network is diffuse in the composites. In addition, the removal of associated cables and wires for linking individual sensors also brings additional merits, and this in particular mitigates the degradation of adhesive layers between the host structure and a sensor network under cyclic loads, and reduces the weight and volume added to the original structures.

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Though only a few PZT wafers are still used as GUW actuators to generate and maintain continuous propagation of probing GUWs in the composites, there are, in principle, an infinite number of sensing paths that can be created at the percolating sensing network, and GUWs can be captured whithersoever. Such a sensing philosophy is illustrated schematically in **Figure 5**. Printed electrical circuits and electrodes can further be used to network PZT actuators and self-sensing networks. Not only are the composites thus fabricated endowed with significantly enhanced sensing capability with exponentially increased sensing paths, but they also minimize the intrusion from an additional sensor system to the original structures. This addresses a concept of "sensor-free" GUW acquisition, which is crucial to maintain the integrity of original composite structures and eliminate material degeneration due to an added transducer network.

Upon acquisition of GUW signals from the sites of interest, desired signal features can be extracted, either in a linear domain (e.g., time-of-flight (ToF) [53-55], wave reflection and transmission coefficients [56-58], energy dissipation [59, 60] and converted wave modes [61, 62]) or in a nonlinear domain (e.g., first-[63], second-[64] or sub-[65] harmonics, spectral sidebands [66], shift of resonance frequency [67]). Focusing on development and demonstration of a new breed of nano-engineered composites with capability of self-sensing GUWs, this study adopts an easy-to-extract signal feature – ToF – for identifying structural damage, as a proof-of-concept demonstration. In a nutshell, with ToF extracted from a captured GUW signal, the location of damage, if any in the inspected composite structure, can be triangulated via

$$t_{A-D-S} - t_{A-S} = \left(\frac{L_{A-D}}{V_1} + \frac{L_{D-S}}{V_2} \right) - \frac{L_{A-S}}{V_1} = \Delta t \,, \qquad \textbf{(5)}$$

where

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$$L_{A-D} = \sqrt{\left(x_d - x_a\right)^2 + \left(y_d - y_a\right)^2},$$
$$L_{D-S} = \sqrt{\left(x_s - x_d\right)^2 + \left(y_s - y_d\right)^2},$$
$$L_{A-S} = \sqrt{\left(x_s - x_a\right)^2 + \left(y_s - y_a\right)^2}.$$

In the above, $t_{A\text{-}D\text{-}S}$ denotes the ToF of the incident probing GUW propagating from the wave actuator to the damage, and then to the sensing point (any site on the composite structure of interest), and $t_{A\text{-}S}$ the ToF of the probing GUW propagating directly from the actuator to that sensing point. V_1 is the group velocity of the probing GUW, and V_2 the group velocity of the damage-scattered waves (V_1 is not necessarily to be identical to V_2, provided mode conversion occurs upon the interaction of incident probing GUW with damage). $L_{A\text{-}D}$, $L_{D\text{-}S}$ and $L_{A\text{-}S}$ represent the distances between the actuator (x_a, y_a) and the damage center (x_d, y_d), the distance between the damage center and the sensing point (x_s, y_s), and the distance between the actuator and the sensing point, respectively. Δt (i.e., ToF) is to be determined from a captured GUW signal. With known V_1, V_2, (x_a, y_a) and (x_s, y_s), the solutions to Eq. (5) can be ascertained, which mathematically shape an elliptical or an ellipse-like locus with the actuator and the sensing point being two foci – the dot line shown in **Figure 6**, indicating all possible locations of the damage center. With ToF extracted from another sensing path, (x_d, y_d) can be obtained. Note that for the fabricated self-sensing composites, GUW acquisition can be implemented at any sites of the composites, and with more sensing paths the location of the damage can be determined by mathematically seeking the intersection of all loci.

To facilitate the above process, a probability-based diagnostic imaging (PDI) algorithm [52, 60, 68, 69] is introduced, able to visualize the identified damage location in a two-dimensional greyscale image. PDI presents the diagnostic results in terms of the probability of presence of damage in an inspected structure. Using PDI, the inspection region of the composite structure is meshed virtually, and projected to an image with each image pixel

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corresponding exclusively to a spatial point in the inspected region. The probability of damage presence at each spatial point is then calibrated in terms of the value borne by its corresponding pixel in the image, via

$$S(\mathrm{x}, y) = \sum_{r=1}^{n} A_r(x, y) D_r \left(\frac{\sqrt{(x - x_a^r)^2 + (y - y_a^r)^2} + \sqrt{(x - x_s^r)^2 + (y - y_s^r)^2}}{V} \right), \quad \textbf{(6)}$$

where $S(x, y)$ denotes the field value at location (x, y) for the r^{th} sensing path in the sensing network, which is linked to the probability of damage occurrence therein. $D_r(t)$ signifies the profile of energy amplitude of the relative difference between the baseline and current signals, captured via the r^{th} path, which reads

$$D(t) = \frac{H(t) - H_0(t)}{H_0(t)}, \quad \textbf{(7)}$$

where $H(t)$ and $H_0(t)$ are the Hilbert transform-processed current signal and baseline signals, respectively. $A_r(x, y)$ represents a weight coefficient determined by the propagation distance of the r^{th} path as

$$A(x, y) = \left[\beta - \left(\frac{\sqrt{(x - x_a)^2 + (y - y_a)^2} + \sqrt{(x - x_s)^2 + (y - y_s)^2}}{\sqrt{(x_a - x_s)^2 + (y_a - y_s)^2}} \right) \right] / (\beta - 1), \quad \textbf{(8)}$$

where β is a scaling parameter controlling the size of the elliptical locus area that is 1.05 in this study (see [70] for selection criteria of β). Again, the present study accentuates development of functionalized composites with self-sensing of GUWs, and details of the signal processing and damage identification algorithms can be referred to authors' previous work [69-72].

4. Experimental Validation

4.1 Description of System and Samples

A system for generating and acquiring GUWs is developed, illustrated schematically in

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Figure 7. The system comprises three core modules: i) GUW generation module (consisting mainly of an arbitrary waveform generator (NI® PXI-5412) and a linear power amplifier (Ciprian® US-TXP-3)), via which tailor-made probing GUWs in narrow-band waveforms can be generated in a frequency range of 0-2.5 MHz; ii) GUW acquisition module (consisting mainly of a digitizer (NI® PXI-5105) and a signal conditioner (Ciprian® LNA-EO-3)), whereby GUW signals can be acquired at a sampling rate up to 60 MHz; and iii) a central control and post-processing module embedded with in-house software interface. In particular, a self-developed, dedicated signal amplifier is integrated in the system, to facilitate acquisition of GUWs of high frequencies yet ultralow magnitudes. The amplifier converts resistance signals to voltage signals upon noise filtering.

Using the manufacture procedure described in Section 2.2, an 8-layer [0/90] laminated composite plate made of plain-woven fabrics, measuring 300 × 300 × 1.4 mm^3, is fabricated, with a diffuse graphene nanoparticle-formed percolating sensing network. The laminate, in its intact status, is used to examine its capability of self-sensing GUWs. The self-sensing is implemented by painting a silver electrode pair with a gap of ~1 mm on the composite surface at any desired sensing location. This gap is prudently selected and controlled during fabrication, to achieve an electrical resistance that is compatible with that of the Wheatstone bridge in the signal amplifier.

4.2 Frequency Sweep Test

To examine the correctness and sensitivity of the fabricated composite laminate to self-perceived GUWs of different frequencies, 5-cycle *Hanning*-function-modulated sinusoidal tonebursts are emitted into the laminate at a central frequency ranging from 50 kHz to 500 kHz with an increment of 10 kHz, using the system as described in Section 4.1. The

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generated toneburst signals drive four PZT wafers (Haiying Enterprise Group Co., Ltd., P-51, 12 mm in diameter and 1 mm in thickness) that are surface-mounted on the laminate, see **Figure 7**. Acquisition of GUWs is implemented at multiple sensing points of the laminate that are arbitrarily selected. For the purpose of comparison and calibration, a multitude of PZT wafers (the same as those as the actuators) are used, each of which is collocated next to a selected sensing point to capture GUWs in synchronism.

4.3 Results and Discussions

Without loss of generality, **Figure 8** shows representative signals i) captured via a sensing path, linking a PZT actuator (75 mm from the left edge and 246 mm from the bottom edge) to the sensing point (75 mm from the left edge and 96 mm from the bottom edge), and ii) simultaneously captured using two PZT wafers that are collocated near the sensing point (5 mm from the sensing point), when the probing GUW signal is excited at 180 kHz. A Butterworth filter is applied to screen measurement noise. In the signals acquired by two PZT wafers and self-perceived by the composite laminate itself, the first-arrival wave component (viz., the zeroth-order symmetric Lamb wave mode guided by the laminate, denoted by S_0 hereinafter) are clearly observed to dominate signal energy. Signals are in good and quantitative agreement, in terms of the arrival time in particular. The main difference between signals measured by PZT wafers and by the self-sensing network is the crosstalk at the initial moment of excitation. This crosstalk is more pronounced in the self-perceived signals than that in the PZT-captured signals – it can be attributed to the intrinsic electrical conductivity of the composites with graphene nanoparticles-formed percolating sensing network. Knowing that crosstalk-related noise occurs at the onset of each signal acquisition, it can be removed via proper signal processing.

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Figure 9 shows the spectra over the time-frequency domain of the signals self-captured at the sensing point same as the above, with the frequencies of the probing GUWs varying from 50 kHz to 500 kHz, which are compared against the spectra of counterpart signals captured with PZT wafers nearby. Reasonably good agreement between two spectra is noted, in which both the S_0 mode and the zeroth-order anti-symmetric Lamb wave mode (denoted by A_0, with a lower propagation velocity) are captured, showing that the amplitude of S_0 mode reaches its peak at an excitation of ~180 kHz. Such a phenomenon – the frequency-dependent signal amplitude, is referred to as *wave mode tunning* [73]. Note that the difference in signal magnitude captured by PZT sensors and by graphene nanoparticle-based sensing network is attributed to their completely different sensing mechanisms: a PZT wafer measures the piezoelectricity which is most sensitive to surface strain, whereas the graphene-based sensing network perceives piezoresistivity that reflects the strain change through the entire waveguide thickness.

Signals captured at other locations of the laminate all present good agreement with counterpart signals obtained with nearby PZT wafers, demonstrating that the graphene nanoparticles have formed a uniform, even and percolating sensing network in the functionalized composite laminate, and GUW propagation in the laminate can be monitored at any desired location of the structure – addressing the concept of "sensor-free" SHM.

5. Proof-of-Concept Application: SHM of Composite Laminate

Another composite laminate that is identical to the one examined in the above experimental validation is prepared following the same manufacture process, in which the graphene nanoparticle-formed percolating sensing network is created. The condition of the pristine laminate is first inspected using infrared (IR) thermography, showing no observable

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fabrication defect in the sample. During IR thermography scanning, hot air flow is used to provide thermal stimulation to the sample, and the thermal radiation emitted from the surface of the sample is sensed and recorded with a commercial thermographic camera (NEC® TH9100PMV).

A drop-weight test is performed to introduce barely visible impact damage (BVID) in the laminate. Subsequent IR thermography shows the BVID in the laminate, as seen in **Figure 10**. A PZT wafer (Haiying Enterprise Group Co., Ltd., P-51, 12 mm in diameter, and 1 mm in thickness) is surface-mounted on the laminate, 75 mm from the left and 246 mm from the bottom of the laminate, to generate probing GUW signals. GUW propagation in the laminate (upon interaction with the BVID) is self-monitored by the laminate in conjunction with the system described in Section 4.1. Based on the frequency sweep test (Section 4.2), the amplitude of the S_0 mode is observed to dominate signal energy at ~180 kHz, and is halved when the excitation frequency lower than 140 kHz or greater than 260 kHz (i.e., *wave mode tunning*). Therefore, the S_0 mode at 180 Hz is chosen as the probing GUW for BVID characterization in pursuit of a high signal-to-noise ratio. The group velocity of the S_0 mode at 180 Hz is ascertained in terms of the arrival time in **Figure 8**.

With the ToF-based PDI, as elaborated in Section 3, the BVID is characterized and visualized in a diagnostic image, **Figure 12**, based on the measurement at four sensing points of the laminate ((75, 96), (160, 16), (274, 40) and (274, 165), as shown in the **Figure 11**), showing good agreement with the reality. In the present study, for the purpose of demonstration, only several sensing points are arbitrarily selected, at each of which a pair of silver electrodes is painted therein for transferring captured GUW signals to the signal acquisition system. However, it is noteworthy that GUWs propagating in the functionalized laminate can be self-

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sensed by the laminate at any site – that is because the sensing capability of the functionalized composites is guaranteed by the fully dispersed nanoparticles in the polymer matrix. Thus, the electrodes can be painted at any sites of the laminate, from which GUW signals can be obtained.

6. Concluding Remarks

This study explores the self-sensing capability of a new breed of nano-engineered functional composites, in which a 2-D graphene nanoparticle-formed percolating self-sensing network is diffused in the dielectric polymer, and the measured piezoresistivity is linked to the dynamic strain up to 500 kHz induced by propagating GUWs. The transient dynamic strain causes disturbance to the sensing network and changes the local electrical resistance accordingly. The change in resistance is then converted into voltage signals to construct wave responses. The experiment results have revealed that GUW signals can be captured precisely by the percolating self-sensing network, comparable to conventional PZT sensors. With self-sensed high-frequency GUW, *in situ* health monitoring can be implemented for composite structures.

The developed self-sensing approach features merits over conventional sensing means adopted in GUWs-based SHM such as PZT transducers. The sensing of GUWs can be performed at any desired position of the composite structure – a trait that is substantially distinct from conventional measurement in which GUWs can only be captured by fixed sensors at specific locations. The significant reduction in the use of cables and wires for linking sensors mitigates the degradation of adhesive layers between the host structure and a sensor network under cyclic loads, and reduces the weight and volume added to the original structure. Together, this makes a dense sensing network be formed in conventional fibre-

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reinforced polymer composites, providing a cost-effective solution for "sensor-free" SHM. Due to the fully dispersed graphene nanoparticles in the polymer matrix, the self-sensing capability of the functionalized composites is available throughout the entire laminate, addressing a concept of "sensor-free" SHM. For demonstration purposes in this study, only few sensing points are arbitrarily selected to acquire GUW signals. In future development, printed electronics-based circuits will be introduced to improve the current methodology and get a step closer to full "sensor-free" SHM.

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