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A Coatable, Lightweight Nanocomposite Sensor for *in-situ* Acquisition of Ultrasonic Waves and Its Application to Embeddable Structural Health Monitoring

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Abstract

Lightweight and resilient, a nano-engineered sensor was developed, coatable to engineering structures via a screen printing approach, for in-situ acquisition of ultrasonic waves for implementing guided ultrasonic wave (GUW)-based structural health monitoring (SHM). Carbon black (CB)/polyvinylidene fluoride (PVDF)-based hybrid with various degrees of percolation were prepared, to fabricate the sensor. In an ultrasonic regime, GUW modulates the infrastructure of formed conductivity network of CB nanofillers with introduction of tunneling effect, and consequently changes the piezoresistivity manifested by the sensor. Morphological characterization, and static/dynamic electro-mechanical response tests were conducted to ascertain an optimal percolation threshold of the conductivity network. At the optimal threshold (~ 6.5 wt%), the sensor exhibits high-fidelity, fast-response, and high-*sensitivity to GUWs up to 400 kHz. Addressing an innovative sensing philosophy of "quasi-dispersed sensing", the sensor presents a potential to strike a compromise between "se*nsing *cost" and "sensing effectiveness", well accommodating the needs from GUW*-based SHM.

1 INTRODUCTION

The use of conductive polymer composites (CPCs) for developing new sensors has been increasingly explored [1-4]. Representative nanofillers of CPCs include carbon nanotubes (CNTs) [1, 2], carbon black (CB) [5], carbon fibers [6], and graphene [7], to name a few. Owing to their nano-scale sizes, the nanofillers are able to be dispersed in polymer matrices uniformly. CPCs bear some appealing and unique material features, such as low density, desired flexibility, easy tailorability in shape, chemical stability, and favorable electrical-mechanical performance. Recently, based on the demonstrable capability of perceiving structural and ambient changes with desired sensitivity and accuracy, CPCs-based sensors have been successfully deployed for various paradigms, such as detection of gas leakage and content [8], measurement of static or quasi-static strains [1], acquisition of dynamic responses [9], and detection of damage [10].

A great amount of effort has been devoted to the development of CPCs-based piezoresistive strain sensors for measuring static (or quasi-static) deformation [11], on which basis a diversity of applications, and damage detection in particular, can be implemented. In most applications, damage can be indicated through the change of electrical conductivity of the sensor which is adhered to the host structure. Representatively, CPCs-based piezoresistive sensors composed of CNTs and polyvinylidene fluoride (PVDF) were prepared and tested regarding performance of stability and sensitivity with various CNT concentrations [12]. In another example, multi-walled CNT thin films were fabricated by a layer-by-layer (LbL) technique [13]. The responses of the thin films to various stimuli were investigated, whereby a two-dimensional mapping of the conductivity of CNT thin films was constructed. The results are conducive to the development of a novel sensing skin. As an extension of the above work, CNT thin films were spray-deposited directly on a glass fiber composite laminate [28], and the spatially distributed electrical conductivity of the films was monitored, indicating a quantitative impact monitoring diagnosis via an electrical impedance tomography algorithm. Compared with conventional metallic foil-based strain gauges, CPCs-based piezoresistive strain sensors have been demonstrated more sensitive to material deformation, with additional merits such as enhanced measurement repeatability.

Though effective in detecting the existence of damage, most CPCs-based piezoresistive sensors, by measuring damage-caused changes in electrical resistance (ER), are only capable of perceiving a relatively large material deformation (of the order of millimeter) when the material undergoes a static or quasi-static (few hertz) load, and thus, are usually difficult to quantitatively characterize damage. Contrarily, guided ultrasonic waves (GUWs), featuring an extremely small ultrasonic elastic disturbance (of the order of micrometer), has shown its successful applications in quantitative damage detection [14, 15].

Motivated by this and enabled by recent advances in nanotechnology, sensor technique, and electronic packaging, a nano-engineered, lightweight CPCs-based nanocomposite sensor made of a hybrid of CB and PVDF was developed in this study, for implementing damage detection and structural health monitoring (SHM) which take advantage of GUWs. In the sensor, CB nanofillers serve as the reinforcement, while PVDF as matrix, to optimally form a conductive network. Electrical analysis, morphological characterization, and quasi-static electro-mechanical response investigation were performed, to advance insight into the tunneling effect in the formed conductive network when the sensor is in response to dynamic elastic disturbance. An optimal percolation threshold of the CB network was ascertained and selected, in order to achieve favorable sensitivity to high-frequency strain represented by GUWs in the order of 10^5 Hz and several microstrain. To examine the correctness, sensitivity and accuracy of the fabricated nanocomposite sensor, captured GUW signals were compared with the counterpart signals acquired by conventional piezoelectric wafer. Featuring the merits of coatability to host structures and deploy ability in a large quantity to configure a dense sensor network, this lightweight nanocomposite sensor is conducive to in-situ sensing towards SHM.

2 SENSOR FABRICATION AND CHARACTERIZATION

2.1 Material preparation

CB (N220, with an average particle diameter of 80 nm, supplied by CABOT Corporation) was chosen as the nanofiller, while PVDF (Kynar k721, with a density of 1.74 g/cm^3 and a melting point of ~158 ºC, supplied by ARKEMA) as the matrix, to fabricate a nanocomposite hybrid. Twofold considerations lead to the above choice: (i) CB has a nano-scale size and high specific surface area compared with other nanofiller candidates such as CNTs and, it also features much reduced amount of entanglement of nanoparticles, facilitates the formation of an even, stable and uniform conductive network; (ii) PVDF is able to response faster to dynamic change compared to traditional rubber-based piezoresistive materials (rubber-based often exhibiting complex time- and frequency-dependent viscoelastic properties, restricting them from being responsive to fast-change signals). In addition, PVDF possesses other merits including low-density, chemical inertness, thermal stability, easy-processing, and flexibility. Altogether, CB and PVDF endow the fabricated nanocomposite hybrid with enhanced sensitivity and high-fidelity response to dynamic changes applied to the formed conductive network.

CB nanofillers were dispersed into PVDF through a standard melt-mixing process in an internal mixer at 190 ºC and 50 rpm for 0.5-hour. Several mass ratios of CB nanofillers to PVDF in the hybrid, ranging from 5 to 30 wt%, were considered during preparation for performance optimization. The fabricated nanocomposite hybrid was immobilized between two steel plates and molded to films ~200 μm in the thickness, using a standard hot-pressing process in which each film was hot-pressed at 20 MPa and 190 ºC for 10-minute, and then cooled to a room temperature at an atmospheric pressure for another 60-minute. A photographic picture of the film as shown in Figure 1(a) indicates the high resilience of the fabricated sensor. Upon curing, the nanocomposite films were tailored to rectangular flakes, each measuring 10 mm × 6 mm (thickness remained at ~200 μm). To introduce electrodes to each flake to configure a sensor, each flake was silver pasted using a dual-component (polymer/silver powder) adhesive (D05001, Beijing Emerging Technology Co. Ltd., China) with shielded cables. A sensor with two electrodes in its final fashion is photographed in Figure 1(b). Small and lightweight, such a sensor can be produced in a single fabrication process, and deployed in a large quantity for configuring a sensor network.

(a) (b)

Figure 1 (a) The photographic image of the hybrid CB/PVDF and (b) a fabricated CB/PVDF nanocomposite sensor with silver pasted two electrodes

2.2 Material characterization and quasi-static testing

Conductivity, as an essential indicator of sensor performance, was examined first. ER of each sensor at various CB weight ratios (5, 6.5, 8, 10, 20 and 30 wt%) was measured using a standard four-probe method with a semi-conductor characterization system (4200-SCS, Keithley Instrument, Inc., Cleveland, Ohio, the USA). With ascertained ER (R), the electrical conductivity (denoted by σ) was defined by $\sigma = l/(R \times A)$, where A and l are the effective area and length between two electrodes of a sensor, respectively. The accordingly obtained average σ (Siemens per meter, S/m) are plotted in Figure 2. A conspicuous increase in electrical conductivity of the hybrid by a six-order of magnitude can be observed from 5 wt% to 8 wt%, this indicating the formation of a fully conductive CB network in the sensor at this particular CB weight ratio, which is deemed to be in a vicinity of the percolation threshold. The percolation threshold [16] represents a transition of the hybrid from insulation to conduction, beyond which a slight increase in CB content can give rise to a tremendous leap in the number of conductive paths formed by dispersed CB nanofillers and consequently an abrupt increase in conductivity of the sensor.

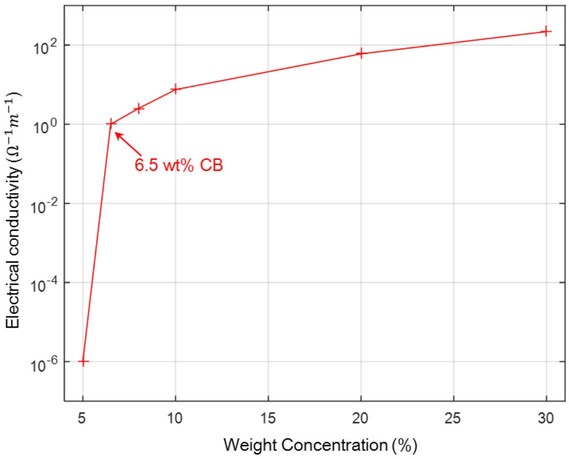


Figure 2 Average conductivity (σ) of the fabricated CB/PVDF nanocomposite sensors at different CB weight ratios

A further morphological characterization was implemented on the hybrids using a scanning electron microscopy (SEM) (JSM-7500F, JEOL Ltd.) to interrogate the sensing mechanism. The obtained SEM image, at the optimal CB weight ratio of 6.5 wt%, is displayed in Figure 3. It can be observed that CB nanofillers are dispersed in PVDF randomly and evenly, this implying a good fabrication process of the sensor. In particular, at the determined percolation threshold (~6.5 wt%), it is apparent that a substantial number of conductive paths have been created in the CB network owing to dramatically increased connections among CB nanofillers and their aggregations. This yields an instantaneous transition from insulative to conductive status of the sensor.

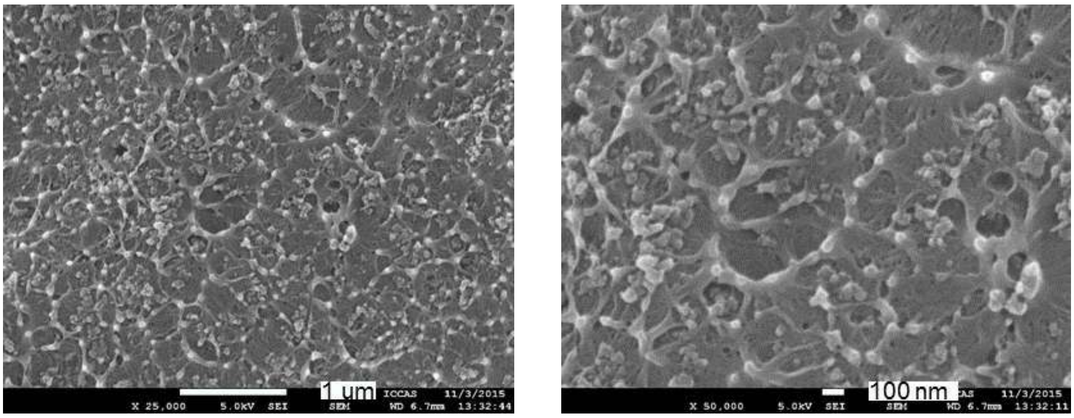


Figure 3 Nano-scalar structures of the fabricated CB/PVDF nanocomposite sensors at the CB weight ratio 6.5 wt% (right: zoomed-in SEM image of the one on left)

Residing on the above ascertainment of the piezoresistivity mechanism due to tunneling effect, the sensor was finalized for strain sensing. A cyclic load windowed in a ramp strain mode with a ramp rate of 1.0%/min was applied until reaching its maximum at 1.0%. As a representative result, the change ratio of ER measured by the sensor at the percolation threshold (~6.5 wt%), when subjected to the applied cyclic load, is displayed in Figure 4, to observe a consistence between the response and the applied cyclic load without discernable hysteresis, this asserting good dynamic stability and desirable measurement repeatability of the sensor.

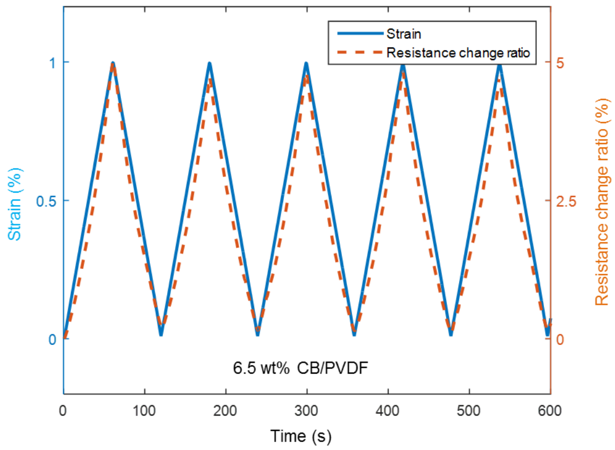
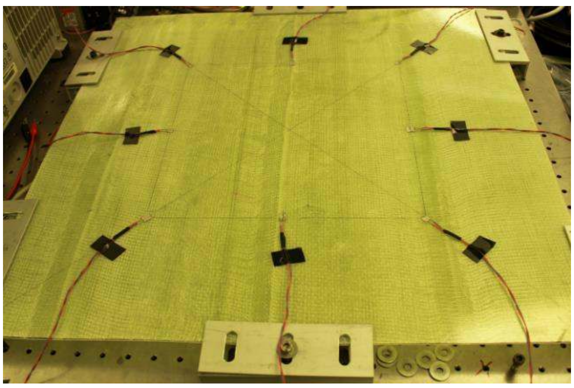
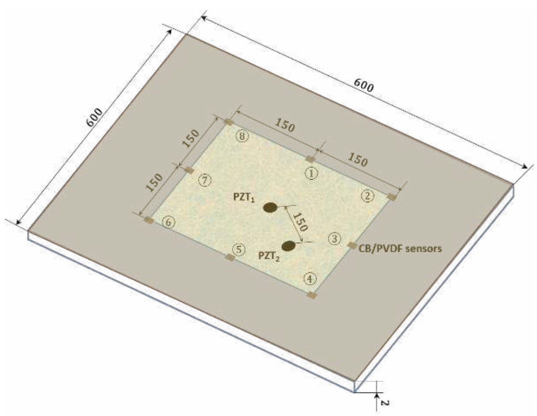


Figure 4 Electro-mechanical response of the fabricated CB/PVDF nanocomposite sensors in comparison with applied cyclic load (at percolation threshold)

3 ACQUISITION OF HIGH-FREQUENCY GUWS

Based on the above material characterization and quasi-static testing, performance of the sensor was further examined in an ultrasonic regime. A glass fiber-epoxy composite laminate panel (600 mm × 600 mm × 2 mm) was prepared, as shown in Figure 5(a). To activate GUWs into the panel, two PZT wafers, denoted by PZT_1 and PZT_2, respectively, were surface-mounted on the laminate with their respective locations specified in Figure 5(b) as transmitter and receiver, respectively. Eight nanocomposite sensors were surface-glued on the laminate, on the same side of the laminate as two PZT wafers, to form a sensor network with a coverage area of 300×300 mm^2.





(a) (b)

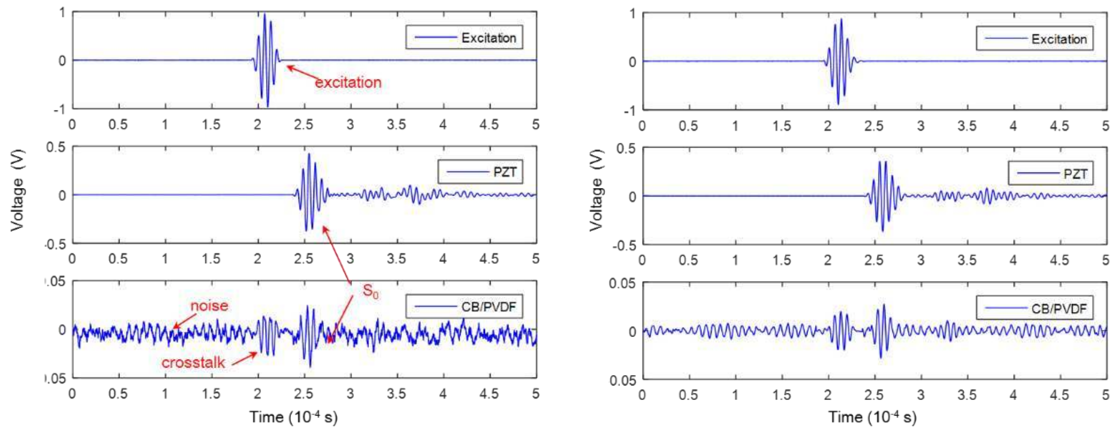
Figure 5 (a) Photographed and (b) schematic of the experimental set-up for acquisition of high-frequency

GUWs propagating in a glass fiber-epoxy composite laminate panel (unit: mm)

Five-cycle Hanning-windowed sinusoidal tonebursts at a central frequency varying from 50 to 400 kHz were generated by the waveform generator, amplified to 400 Vp-p via the linear power amplifier, and then applied on PZT_1 to excite GUWs. The generated GUWs propagating in the laminate were acquired by the eight sensors and PZT_2 through the oscilloscope at a sampling rate of 10 MHz.

As a result of weakness of the magnitude of GUWs in a frequency range of kilo- or mega-Hertz (GUW-induced elastic disturbance is usually of the order of several micro-strains), the represented ER change ratio of the sensors may be extremely faint. To avoid a low signal-to-noise ratio, a self-contained signal amplification and acquisition system was developed, to be used in matching with the sensors. The system involves a signal amplification module and a signal generation/acquisition module developed on a PXI (PCI eXtensions for Instrumentation) bus platform (NI® PXIe-1071).

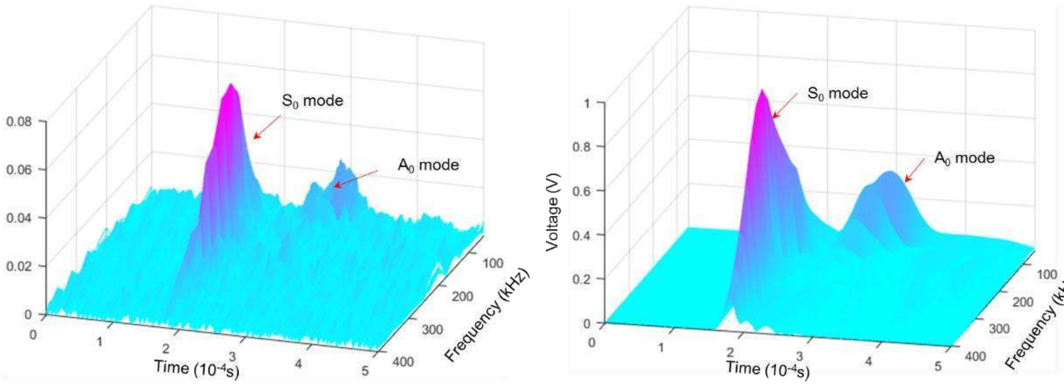
The amplification module integrates a Wheatstone bridge with resistors compatible to the ER of the selected sensor (ER was ascertained via electrical analysis detailed in Section 2.2), an electronic amplifier circuit, a series of filters and a signal conversion unit (for converting measured piezoresistivity to electrical voltage signals). The signal generation/acquisition module consists of a waveform generator (NI® PXIE-1071) (for exciting GUWs via PZT wafers (Physik Instrumente Co., Ltd., PIC151; diameter: 9 mm; thickness: 0.5 mm)), a linear power amplifier (Ciprian® US-TXP-3), and an oscilloscope (Agilent® DSO9064A). Using these two modules, GUW signals were generated and then captured.



(a) (b)

Figure 6 (a) Raw and (b) filtered signals at 150 kHz (from top to bottom: excitation signal and response signals acquired by PZT and CB/PVDF)

Illustratively, Figure 6 shows the raw and filtered GUW signal (first-order Butterworth filter) captured by nanocomposite sensor ① (see Figure 5), against the counterpart signal collected by PZT_2, when PZT_1 served as the actuator to generate GUWs at 150 kHz. In the figure, the first-arrival wave components (viz., the zeroth-order symmetric Lamb wave mode guided by the laminate, denoted by S_0) perceived by both the nanocomposite sensor and PZT wafer are observed to be coincident in terms of the arrival time. In addition, all the nanocomposite sensors in the sensor network show high consistence in performance in the discussed frequency range (50~400 kHz), as shown in the mesh plot in Figure 7, The difference in respective magnitudes can be attributed to the distinguishably different sensing mechanisms of the two types of sensors: PZT wafer is a piezoelectric sensor, while the nanocomposite sensor is a piezoresistive sensor by exploring variation in a formed CB conductive network with induced tunneling effect. The comparison implies the independence of the piezoresistive behaviors evidenced by the sensor on the frequency of dynamic elastic disturbance.



(a) (b)

Figure 7 Magnitudes of GUWs (50kHz ~ 400kHz) acquired by (a) CB/PVDF sensor ① and (b) PZT$_2$

4 FURTHER DEVELOPMENT

Improved damage detection and SHM ability can be realized with spatially distributed sensors by forming a networked communication with each other. However, the “sensing cost”, i.e., the weight and volume penalty to the host structure, and the “sensing effectiveness”, i.e., the information redundancy to improve detection accuracy and correctness, present an interior contradiction. For instance, a sensor network comprised of a limited number of distributed sensors (such as PZT) is likely to “overlook” information, and deliver inaccurate or even erroneous results. However, the developed nanocomposite sensor, lightweight and small, can be coated in a large quantity to a structure with least weight and volume penalty to form a dense sensor network. Such a “quasi-dispersed sensing network” allows acquisition of rich information that would be very difficult to achieve using a conventional network with sparsely distributed sensors (e.g., PZT). Diverse flexible sensor networks can be configured with advanced processing or molding methods conveniently and quickly. Besides, this flexibility is not restricted by the material (metals or composites) or geometric (flat or curved) traits of the host structure to be coated. Featuring these merits, the developed nanocomposite sensor has a potential to achieve a desirable balance between “sensing cost” and “sensing effectiveness”.

5 CONCLUSIONS

A lightweight nanocomposite sensor made of a hybrid of CB/PVDF was developed, to fulfill in-situ acquisition of dynamic elastic disturbance of ultrasonic waves up to 400 kHz. Via rigorous material characterization including electrical, morphological and quasi-static electro-mechanical response interrogation, the nano-scalar architecture of the sensor was optimized to reach a desired percolation threshold of the CB conductive network (~6.5 wt%), which has been further demonstrated sensitive to dynamic elastic disturbance in an ultrasonic regime up to ~400 kHz with a magnitude as low as in the order of microstrain. This study has revealed that the tunneling effect among nanofillers dominates the sensing mechanism of the sensor in perceiving dynamic particulate motion. A frequency-independent piezoresistive behavior is also testified. In conjunction with a dedicated signal amplification apparatus, the feasibility, correctness and sensitivity of the sensor was examined, against conventional piezoelectric transducers, to demonstrate excellent coincidence between two types of sensors. Coatable to a structure and deployable in a large quantity, this new type of nanocomposite sensor has blazed a trail for implementing in-situ sensing for ultrasonic wave-based damage detection and SHM, by striking a compromise between “sensing cost” and “sensing effectiveness”.

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