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Graphene-based Nanocomposite Strain Sensor Response to Ultrasonic Guided Waves

Feng Duan[1,3§‡], *Yaozhong Liao*[2§‡], *Zhihui Zeng*[1,4], *Hao Jin*[1*], *Limin Zhou*[2], *Zhong Zhang*[1*] *and Zhongqing Su*[2*]

[1]CAS Key Laboratory of Nanosystem and Hierarchical Fabrication, CAS Center for Excellence in Nanoscience, National Center for Nanoscience and Technology, Beijing 100190, P.R. China

[2]Department of Mechanical Engineering, The Hong Kong Polytechnic University, Kowloon, Hong Kong SAR

[3]University of Chinese Academy of Sciences, Beijing 100049, P.R. China

[4]School of Materials Science and Engineering, Nanyang Technological University, 50 Nanyang Avenue, Singapore 639798, Singapore

[*]E-mail: hjin@nanoctr.cn; zhong.zhang@nanoctr.cn; zhongqing.su@polyu.edu.hk

[§] These authors contributed equally to this work.

[‡] PhD students

Abstract: Certain traditional sensors like lead zirconate titanate (PZT) wafers and ultrasonic probes can respond to extremely weak disturbances such as ultrasonic guided waves (UGWs). However, their further development for applications to meet increasing engineering demands is limited on account of their hardness, brittleness, and complex manufacturing processes. Now, emerging nanotechnology ushers in a brand-new world

for nanocomposite-based strain sensors, endowing them with higher flexibility, better surface compatibility and easier fabrication. Yet there are few reports of composites which can be used to perceive high frequency UGWs with an ultralow magnitude. Here, we present a novel graphene-based nanocomposite possessing strong sensitivity for sensing ultrasonic waves by virtue of a neoteric sensing mechanism – the tunneling effect. By designing and optimizing the microstructure of the conductive network in the nanocomposite sensor, we successfully capture ultrasonic waves with high signal-to-noise ratio in a broad frequency range up to 1 MHz. With the feature of high sensitivity and rapid response times, the graphene-based nanocomposite becomes a promising candidate for structural health monitoring in developing prospective applications.

KEYWORDS: graphene-based nanocomposites; flexible strain sensor; ultrasonic guided waves; piezoresistivity

1. Introduction

Flexible sensors, with outstanding compatibility to arbitrarily curved surfaces, have attracted considerable attention in recent years for the developing interests of smart robots, wearable devices and flexible electronics [1-5]. In particular, flexible strain sensors have been a focus for promising potential applications in human health monitoring [2, 6-10] and structural health monitoring [11-13]. Conventional strain sensors made of ceramics and metals suffer from intrinsic hardness, brittleness, and complex manufacturing processes, and can hardly satisfy the demand for fast-developing engineering applications [12]. The burgeoning development of nanomaterials and nanocomposites has blazed a trail in the manufacture of flexible

strain sensors. Nanocomposite strain sensors, possessing fascinating properties such as light weight, low cost and high flexibility, can be easily fabricated and can have high sensitivity by optimization of the synthesis process and selection of a suitable nanofiller [14-16].

Graphene, a one-atom-thick two-dimensional (2D) material with exceptionally high electrical conductivity and excellent mechanical properties, has been widely studied for potential applications since its discovery [17-19]. An important application is the strain sensor based on graphene or its composite because of its high carrier mobility [20] and good piezoresistivity [21, 22] in addition to its outstanding mechanical properties. To date, much effort has been devoted to abtaining graphene-based strain sensors with large stretchability, a broad strain range and high sensitivity, for applications in electronic skins, human health monitoring and wearable electronics [16, 23-30]. For example, Liu *et al.* fabricated a strain sensor with a fish-scale-like graphene-sensing layer, endowing it with a wide sensing range (0.1% ~ 82% strain), high sensitivity (gauge factor of 16.2 to 150), and full-range detection of human motions [28]. A graphene textile-based strain sensor fabricated by Yang *et al.* showed a distinctive negative resistance variation with increasing strain, despite its high sensitivity and close-fitting characteristics [31]. Pan *et al.* designed a 3D graphene/ polydimethylsiloxane hybrid film with a workable strain range up to 187% and simultaneously a gauge factor up to ~ 1500 [32]. A viscoelastic graphene nanocomposite fabricated by Boland *et al.* also exhibited high sensitivity with gauge factors > 500 that could measure pulse, blood pressure, and even the impact associated with the footsteps of a small spider [16]. Yet most reported graphene-based strain sensors, fabricated by various methods and resulting in diverse modalities, are limited

in their ability to measure strain at low frequency (< 200 Hz), as required for the applications to human motion monitoring or wearable electronics.

Nevertheless, ultrasonic guided waves with frequencies as high as hundreds of kilohertz (kHz), as commonly used in structural health monitoring, are still beyond the range of graphene-based strain sensors. The primary challenge originates from the tiny deformation induced by ultrasonic waves with ultra-weak energy. Various strategies have been developed in pursuit of high response frequency, such as a graphene woven micro-fabric structure [33], a crack-based platinum (Pt) coating on polydimethylsiloxane (PDMS) [34], a honeycomb-like foam structure of graphene composite [35], and a graphene/graphene oxide hybrid structure [36], among which flexible strain sensors with a graphene and cellulose elastomer foam structure or with a graphene/graphene oxide hybrid structure can be used to perceive high-frequency vibrations up to 2 kHz and 10 kHz, respectively.

In this work, an innovative strain sensor, manufactured by a fast-prototyping solution-mixing method based on a graphene/ polyvinylidene fluoride (PVDF) hybrid nanocomposite film, is presented, on the basis of our previous discovery – a carbon black sensor that can respond to UGWs up to 400 kHz [12, 13]. The sensing ability results, obtained from a serious of systematically tests under different conditions reveal that the developed sensor, with optimized graphene content, displays high sensitivity, fast response, and high signal-to-noise-ratio (SNR) in response to broadband ultrasound up to 1 megahertz (MHz), with deformation in the scale of nanometers. Featuring a distinct sensing mechanism made from conventional lead zirconate titanate (PZT)-based piezoelectric ultrasound transducers, the response of our graphene-based sensor shows outstanding performance in perceiving UGWs with a frequency much higher than 400 kHz.

2. Experimental

2.1. Preparation of Materials

PVDF powder (Kynar k721, density: 1.74 g/cm^3, melting point: ~158 ºC, ARKEMA Corporation) was dissolved in 1-methyl-2-pyrrolidinone (NMP) (boiling point: 203 °C, XILONG SCIENTIFIC Corporation) with content of 20 wt% at 70 ºC under slow stirring for 3 h. Then graphene aqueous suspension (weight percentage: 2.7%, conductivity: 1× 10^5 S/m, CARBON VALLEY) was added to the PVDF solution and the mixture was stirred by a low-speed mixer for 2 h at the speed of 800 rpm. The mixture was then degassed in a vacuum oven at 60 ºC for 30 min to remove trapped air bubbles, after which, was poured onto glass board and cured at 150 °C for 2 h. After cooling in air, the composite ~200 μm thick film was obtained by peeling it from the glass board. To be used as a functional sensor, the composite film was cut into tailored pieces, each of which was affixed with shielded wires via silver paste as electrodes.

2.2. Characterization

The section morphology of the nanocomposite with various graphene contents was observed by scanning electron microscopy (SEM, JEOL JSM-7500F). X-ray diffraction (XRD) analysis was implemented at room temperature on an XRD platform (X'Pert Pro, PANalytical) with a specular reflection mode (Cu Ka radiation) and the scanning angle varying from 10 ° to 50 ° (with a scanning rate of 4 °/s). The resistance (R) of the nanocomposite was measured by the four-probe method with a semi-conductor characterization system (4200-SCS, Keithley Instruments, Inc.). The electrical conductivity (σ) was calculated by the equation $\sigma = l/(R \cdot A)$, where l and A are the effective length and area of the measuring electrode, respectively. The effective area $A = w \cdot t$, where w and t refer to the width and the thickness of the measuring sample,

respectively, as shown in Figure 1a. Typically, the effective dimension of the measured sample is 5 × 10 × 0.2 mm^3. At least three samples for each concentration of graphene nanocomposite film were measured to obtain the average electrical conductivity.

2.3. Acquisition of UGW-induced Strains

It is worth noting that the deformation induced by UGWs is usually subtle in nanometer scale and vulnerable to the surrounding environment, thus leading to complexity for nanocomposite sensors in directly acquiring the tiny electrical resistance changes caused by UGWs. Therefore, a Wheatstone bridge was utilized in the signal acquisition system to amplify the electric signal, as shown in Figure 1b. The Wheatstone bridge, powered by a ± 10 V constant voltage, was properly designed according to the resistance of the nanocomposite sensor developed. In the Wheatstone bridge, a 1000 Ω slide rheostat cascading with a 500 Ω fixed resistor was used as the measurement arm to maintain a balance of the bridge, while the other two fixed arms were installed with a 1000 Ω completion resistor, respectively. The output resistive signal was transferred into voltage signal after being amplified 1000 times via an operational amplifier circuit (ICL7650) and filtered between 1000 Hz and 1.5 MHz.

In the ultrasonic wave signal acquisition system, a five-cycle Hanning-window modulated sinusoidal at various central frequencies was first generated by a waveform generator (NI® PXIE-1071), amplified by a linear power amplifier (CIPRIAN® US-TXP-3) to 200 Vp-p, and then applied to a commercial PZT wafer (Physik Instrumente Co., Ltd., PIC151; diameter: 9 mm; thickness: 0.5 mm) which had been pasted onto the surface of a glass fiber-epoxy composite plate (600 × 600 × 2 mm^3) as an actuator, so that Lamb waves were generated in the composite plate. The excitation signal frequency was varied from 50 kHz to 1 MHz. To identify the signal and make comparisons

conveniently, commercial PZT and nanocomposite sensor pairs were used to capture the ultrasonic wave signal simultaneously, which was recorded by a digital signal oscilloscope (Agilent® DSO9064A) at a sampling rate of 10 MHz. The resistance change in the nanocomposite sensor was transferred to a voltage signal by this signal acquisition system.

3. Results and Discussion

The nanocomposite consists of graphene flakes with an aspect ratio of ~ 4000 (thickness: ~ 5 nm and diameter: ~ 20 μm) as conductive fillers and PVDF as matrix. In general, the electrical conductivity of conductive polymer composite originates from the conductive network formed by conductive fillers and a tunneling effect occurs between adjacent fillers when they are within a critical tunneling distance [13, 37]. The piezoresistivity of a nanocomposite as a strain sensor is based on the variation of conductive network changes under strain. Therefore, the sensitivity of a strain sensor depends considerably on the conductive network formed by fillers in the matrix and usually reaches its highest value with the graphene content at percolation threshold. To determine the percolation threshold, at which the insulating nanocomposite becomes conductive, a series of nanocomposites with various graphene contents were fabricated and the conductivity is shown in Figure 2. With the increase in graphene content, the conductivity of the nanocomposite undergoes a dramatic increase by four orders of magnitude at around 0.5 wt%, on which basis the percolation threshold of the nanocomposite can be accurately calculated according to the percolation theroy that the nanocomposite conductivity (σ) scales with filler fraction f as

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

where f_c and t are the percolation threshold and exponent, respectively [37]. The good linear relation shown in the inset in Figure 2 indicates that the equation fits the experimental data well with f_c = 0.5 wt% and t = 3.2. The percolation threshold of the graphene-based sensor is much smaller than those of the sensors that were earlier made from other types of carbon nanofillers such as carbon black and carbon nanotubes, as reported in the authors' previous studies [12, 13]. This can be attributed to the larger aspect ratio of the graphene and the solution manufacturing process adopted in the present study. However, it is noteworthy that the percolation threshold can be subject to a variety of factors such as manufacture methods adopted and selection of matrix. The larger exponent reveals a broad distribution of intersheet junction resistance [16, 39]. The distribution of graphene sheets in nanocomposites with varying filler content was investigated by SEM as shown in Figure 3a-f. From the SEM images (Figure 3a,c,e) we can observe the sparse distribution of graphene sheets, which are further clearly identified in magnified views (red circle in Figure 3b,d,f), and the formed loose conductive network which tends to become denser as the graphene content increases from 0.5 wt% to 2 wt%. The density of the conductive network is of great importance to the sensitivity of the nanocomposite strain sensor to ultrasonic waves. The polymer matrix of the nanocomposite endows it with high flexibility (Figure 3g) and hence compatibility to curved surfaces, so that it can be easily tailored to the desired dimension. Typically, the sample dimension for acquiring signal from an ultrasonic wave is 5×10 mm^2 (Figure 3h).

Before beginning to capture ultrasonic wave signals by nanocomposite sensor, we performed XRD to eliminate the possibility of piezoelectric effects caused by the PVDF matrix. The XRD patterns of nanocomposites with different graphene content in Figure 4 show no diffraction peak representing graphene at 2θ of 26.1° except for the peaks of

the PVDF matrix, indicating that the graphene sheets are distributed in the amorphous area of the matrix, resulting in loose conductive networks for the existence of crystal phase [40]. The XRD diffraction pattern shows diffraction peaks at 2θ of 17.6°, 18.5°, 20.0° and 26.6°, corresponding to the faces (100), (020), (100) and (021) of α-phase crystals, respectively. Generally, the main crystal phases of PVDF are α and β, of which only the β phase possesses a piezoelectric effect [41]. Therefore, the captured signals at various ultrasonic wave frequencies in the following discussion originates from the piezoresistivity effect of the α phase nanocomposite without piezoelectric effect.

The lowest-order symmetric Lamb wave mode guided by the plate-like sample (denoted by S_0) is basically dominated by the in-plane movement of particles, resembling the across-the-thickness variation of conventional axial plate waves [42]. Due to that fact that the intensity of the S_0 wave mode is much stronger when compared with other wave modes within the frequency range under investigation, S_0 wave mode was chosen for following discussion. In comparisons of the typical ultrasonic wave signal at 200 kHz acquired by 1 wt% nanocomposite sensor with that of the PZT wafer, we found that the S_0 mode for sensors arrives at the same time (Figure 5a), indicating an ultrafast response of the nanocomposite, without any delay. The signal intensity is also comparable with that of the PZT. Signal crosstalk induced by the interference signal arriving at the same time as the excitation signal is observed, and can be easily eliminated by signal processing algorithms. A bandpass filter based on the Fourier transform analysis was designed, aimed to eliminate the measurement noise and uncertainties such as those from the instrument and ambient fluctuation. In the signal spectrum obtained with Fourier transform, 70% and 130% of the frequency, at which the signal was actively generated, were chosen as the cut-off frequencies of the bandpass filter. Applied with the filter, broadband measurement noise was mitigated

and consequently the response of the sensor was remained. After signal filtering, the signal intensity remained almost constant compared with the original data (Figure 5b). On the other hand, the noise signal intensity could be neglected, reflecting that the response signal originates primarily from the ultrasonic waves.

To further assess the sensitivity of the nanocomposite sensor's response to ultrasonic wave, various excitation voltages were applied at a central frequency of 200 kHz. The response signal captured by the nanocomposite became significantly stronger with the increase in excitation voltage from 0.5 V to 1 V (Figure 6a). Figure 6b depicts the relationship whereby the signal amplitude increases linearly with the excitation voltage, proving that the nanocomposite sensor is sensitive to ultrasonic wave as well as quantitatively responsive to ultraweak structural vibrations. Moreover, the slope of the curve for the nanocomposite is greater than that of the PZT, implying that the nanocomposite is more sensitive in sensing ultrasonic waves. The amplitudes of the two signals show no discrepancy in magnitude despite the distinct sensing mechanisms of the two types of sensor.

The density of the conductive network in the nanocomposite has a remarkable influence on its sensitivity in perceiving ultrasonic waves, as manifested in different signal intensities captured by nanocomposites with different graphene content at the same frequency. The signal intensity captured by the 1 wt% graphene nanocomposite is an order of magnitude higher than that captured by 2 wt% nanocomposite in response to the 200 kHz ultrasonic wave (Figure 7a). When the signal intensities of nanocomposites with different graphene contents are compared, the 1 wt% graphene sensor exhibits the highest sensing ability (Figure 7b). For 0.75 and 1 wt% graphene sensors, the variations in the signal amplitudes, both reaching peak values at 200 kHz, have the same tendency when compared with that shown by the commercial PZT wafer.

For the nanocomposite sensors with graphene contents of 1.4 and 2.0 wt%, little regularity can be found in the signal amplitudes. Once the nanofillers are dispersed uniformly in the host matrix, the average distances between two adjacent nanoparticles vary significantly, from several hundred micrometers in the insulating state to fully connected conductive networks with the increase of graphene content. Hence, the sensitivity of the nanocomposite sensor to ultrasonic waves reached highest level when the average distance was close to the optimal tunneling distance within several nanometers.

To further examine the accuracy of signals captured by the nanocomposite sensor, the attenuation of captured ultrasonic waves propagating in the glass fiber-epoxy composite plate used earlier, was compared against that from theoretical prediction. According to the theory of wave attenuation [42], the relationship between two signal magnitudes that are measured at two points along the wave propagation path can be calculated theoretically as

$$\frac{A(d_1)}{A(d_2)} = \frac{\sqrt{d_2}}{\sqrt{d_1}}, \quad (2)$$

where $A(d_1)$ and $A(d_2)$ are the magnitudes of ultrasonic waves at distances of d_1 and d_2 from the actuator, respectively. To explore the highest response frequency limitation of graphene nanocomposites, signals were captured by nanocomposite sensors at various distances from the actuator, with the results shown in Figure 8. With the increase in excitation frequency, the signal intensities captured by the same sensor become weaker, regardless of the distance from the actuator, such as 4 cm and 2 cm (Figure 8a and b). Moreover, the highest sensing frequency increases when the sensor is closer to the actuator, due to the gradual increase in excitation amplitude.

To gain insight into the influence of distance from actuator, the signal intensity under extremely high frequency (≥ 400 kHz) captured by nanocomposite sensors at various distances is summarized in Figure 9a. The signal intensity exhibits a significant declining tendency with the increase in excitation frequency, regardless of the distance from the actuator, due to the ultraweak magnitude. Moreover, the sensing frequency of the nanocomposite increases from 400 kHz to 1 MHz when the sensor is gradually moved towards the actuator from 8 to 2 cm, a feature which suggests manifold application prospects in structural health monitoring. The amplitude voltage at 400 kHz was extracted to further reveal the quantity relationship between sensing amplitude and propagation distance, as shown in Figure 9b. The linear variation trend between the sensing intensity and $1/\sqrt{d}$ is presented, which obeys the aforementioned attenuation equation (Eq. 2) with acceptable error, testifying to the homogenous dispersion status of graphene flakes in polymer matrix.

The size effect of the nanocomposite sensor on the response signal to ultrasonic waves was also examined, as shown in Figure 10. Nanocomposite sensors 4 mm, 10 mm, and 20 mm in length (the length direction is parallel to that of the wave propagation), mounted at a distance of 3 cm from the actuator, were utilized to reveal the geometrical effect. UGWs with the central frequency of 500 kHz, were acquired by nanocomposites with the given sensor lengths, as shown in Figure 10a. It is interesting to find that the signal intensity shows no simple positive or negative correlation with the increase of sample length. The 10 mm sample captures the weakest signal, whereas the 4 mm sample captures the strongest signal. As shown in Figure 10b, the summarized amplitude voltages extracted from signals captured by the 10 mm samples all represent the lowest signal intensity in the 400 ~ 900 kHz frequency range. The signal intensity demonstrates a similar variation trend to that of the PZT wafer except for some slight

deviations induced by noise in the measured frequency range. The maximum frequency detected by the 4 mm sample is 1 MHz, which is 100 kHz higher than that acquired by the two longer samples. Overall, the 4 mm sensor possesses the broadest frequency range up to 1 MHz, while the 10 mm sensor achieves the weakest signal intensity.

The underlying mechanism of dimension dependence of the response signal was further analyzed according to the tuning effect of transducer size [43]. The sensing signal acquired by the sensor originates chiefly from the variations in the conductive network in the nanocomposites, caused by the in-plane deformation excited by S_0 mode Lamb wave. The periodic deformation contained bulging and contracting through the thickness direction. As shown in Figure 11, when the sensor length is an odd multiple of the half-wavelength, the bulging and contracting occurs alternately, thereby changing the distance between adjacent graphene platelets at nanometer scale and consequently resulting in enhanced signal intensity. If the sample length is an even multiple of the half-wavelength, the co-existence of bulging and contracting neutralizes the distance variation, consequently counteracting the tunneling effect and weakening the signal intensity. The wavelengths of the ultrasonic waves with frequencies ranging from 100 to 1000 kHz propagated in a glass-fiber-epoxy laminate panel are calculated. The relationship between sensor length and wavelength under different frequencies is displayed in Table 1. From the result we find that sensors 4 mm and 20 mm in length are closer to the odd times of the half-wavelength at the specific excitation frequency we analyzed, while the sensors 10 mm in length closer to the even multiples. The counteracting effect can cripple the sensitivity of the 10 mm sensor, resulting in the weakest signal among the three sensor lengths.

4. Conclusions

A graphene-based nanocomposite sensor in a feature of the combination of traditional piezoresistivity and tunneling effect was fabricated, which could respond to ultralow dynamic deformation with an ultrahigh frequency range up to 1 MHz. Compared with a piezoelectric PZT, the sensor showed a sensitive and rapid response to broad band ultrasonic waves with a high SNR. The response signal of the ultrasonic guided waves acquired by the nanocomposite sensor showed frequency dependency, with the same tendency when compared with a traditional PZT wafer. The microstructure of the nanocomposite sensor was modified corresponding to the periodic strain variation, leading to a regular resistance change. With its light weight, flexibility, high sensitivity and rapid response, the graphene-based nanocomposite sensor showed promising potential for future structural health monitoring applications.

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