Significant strain-rate dependence of sensing behavior in TiO₂@carbon fibre/PDMS composites for flexible strain sensors

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Abstract: Carbon fibre (CF) embedded into elastomeric media has been attracting incredible interest as flexible strain sensors in the application of skin electronics owing to their high sensitivity in a very small strain gauge. To further improve the sensitivity of CF/PDMS composite strain sensor, the relatively low temperature prepared TiO₂ nanowire via hydrothermal route was employed herein to functionalize CF. The results showed a significant increase in the sensitivity of the TiO₂@CF/PDMS composite strain sensors which was reflected by the calculated gauge factor. As the prepared TiO₂ nanowire vertically embraced the surroundings of the CF, the introduced TiO₂ nanowire contributed to a highly porous structure which played a predominant role in improving the sensitivity of strain sensors. Moreover, the significant strain rate dependent behavior of TiO₂@CF/PDMS strain sensor was revealed when performing monotonic tests at varied strain rate. Therefore, introducing TiO₂ nanowire on CF offers a new technique for fabricating flexible strain sensors with improved sensitivity for the application of flexible electronics.

Keywords: composites; dependence of strain rate; sensitivity; flexible strain sensors

1 Introduction

Wearable strain sensors capable of detecting the strain with a high sensitivity have been attracting tremendous interest in the application of skin electronics, which can be used for health monitoring, human robotics interaction, and tactile perception [1–5]. Compared to conventional strain sensors made of metal or semiconductors [6], stretchable strain sensors demonstrate a superior property, showing a strain limit (can reach $\geq 50\%$)

with alterable sensitivity [7–9].

However, there remain challenges in achieving the practical applications. For instance, the expense with respect to the complicated processes for designing a structure or pattern in wearable strain sensors makes the cost very high [10]. Moreover, when performing the integration of strain sensors, irregular or rough surface will appear instead of flat surface in thin film sensors [7,11]. Therefore, to meet the practical applications in complicated configuration design, a fibre based strain sensor is of great prospects [12,13].

Fibre strain sensors have the advantages of lightweight, flexibility, and long-lasting, which can be developed through either single fibre, thread, yarn, or

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multiple fibre level components [14,15]. For instance, one-dimensional strain sensing yarn wearable textile sensor made of polyurethane/Ag nanoparticle/graphene-microsheets/silicone encapsulation was introduced to achieve the real-time monitoring of human motions [4]. A flexible strain sensor was achieved from core-spun threads with the integrated electrode and sensing cell, where carbon black/silver paste/poly(sodium-p-styrenesulfonate) and single-walled carbon nanotubes/carbon black were used as electrode and sensing cell, respectively [8]. A coaxial structured sensing component made of thermoplastic elastomer-wrapped carbon nanotube fibres was proposed to be used in deformable cable and wearable textiles [12]. A multifunctional sensor array formed through a woven fabric structure with fibre sensor was designed to reach the great potential of scalable manufacturability of e-textile products [16]. The helically wrapped carbon yarn was put forward to detect motion with low strain level [17]. To serve the application of personal healthcare or activity monitoring, a variety of sensors including physical sensors, chemical sensors, and sensors in reaction to environmental stimuli have been developed [18–20]. Meanwhile, a multilayered carbon nanotube/thermal plastic elastomer composite strain sensor was fabricated, and this kind of fibre sensor showed a sensitivity of 21.3 at a wide strain range of 0–150% [3]. Zhong *et al.* [2] reported a stretchable self-powered fibre based strain sensor by coiling a fibre based generator and a silicone fibre. However, to achieve a largely improved strain sensitivity at a small strain gauge for specific practical application, there still exist some issues to be resolved, where aspects of the evolution of conductive network, surface modification strategy in shaping the sensing property, and relationship of configured structure/property are worthwhile to be further explored.

TiO₂ is a chemically stable semiconducting material with a wide band gap [21]. It is also cost-effective and available with abundant sources. For nanostructured TiO₂, great attention has been paid to study the fabrication of TiO₂ in a variety of forms, such as nanowires, nanorods, and nanoparticles, which is targeted in the application of photo catalysts, solar cells, resistance switching, and gas sensing owing to its desired chemical and physical properties. In addition to the investigation of TiO₂ as surface modifier, ZnO nanowire on the surface of a CF was designed to achieve a hybrid structure by packing with flexible substrate PDMS, this piezoelectric strain sensors demonstrated desired sensitivity under

applied strain, which was attributed to the change in Schottky height owing to the strain induced piezoelectric potential [22].

Herein, TiO₂ nanowires grown on CF offer an easy and cost-effective strategy to fabricate a high-performance stretchable strain sensor for the application of flexible electronics. By introducing TiO₂ nanowires, effects due to the properties of interfaces formed between two materials (TiO₂ and CF) with pronounced characteristics. Moreover, the coating TiO₂ with high aspect ratio offers a highly porous structure and enables the effective tuning of conductive network. On the other hand, semiconductor TiO₂ with an increasing density can cause agglomerated fillers to form percolating pathways, which are effective in tuning the sensing performance in a semiconducting way. Therefore, TiO₂@CF provides a highly porous structure for rationally designing the electrically conductive path in a three-dimensional way. CF not only acts as a supporting conductive path, but also enhances the stability of flexible strain sensors during stretching. Consequently, the TiO₂@CF/PDMS composite strain sensors were prepared and investigated under single loading, repeated loading or unloading, followed by studying their sensing mechanism and performance stability.

2 Experimental

2.1 Materials

CFs were purchased from Toray Industries, Japan. PDMS silicone kit including both base and agent (Sylgard 184, USA) was provided by Dow Corning Incorporation.

2.2 Fabrication

The fabrication process of TiO₂@CF/PDMS composites can be described as follows: Firstly, pretreatment was conducted on the pristine CF by washing in ethyl alcohol and deionized water for a certain amount of time to reduce the effect of polymer organics and trace impurities deposited on its surface. Subsequently, the TiO₂ nanowires were prepared onto the CF to obtain TiO₂@CF via hydrothermal technique, where source materials of 100 mL deionized water, 25 mL hydrochloric acid, and 1.7 mL titanium butoxide were taken to perform the fabrication process under the conditions of relatively low temperature of 150 °C with 3 h. For the detailed fabrication process of TiO₂ nanowires, this can

be referred to our previous research work [23]. After this, PDMS was used to immerse the $\text{TiO}_2@\text{CF}$ to achieve the final $\text{TiO}_2@\text{CF}/\text{PDMS}$ composites. Figure 1 shows the detailed fabrication process of $\text{TiO}_2@\text{CF}$, where hydrothermal approach was taken to achieve this kind of coated CF. Subsequently, by using this prepared surface functionalized $\text{TiO}_2@\text{CF}$, PDMS was employed as the elastomer matrix to prepare $\text{TiO}_2@\text{CF}/\text{PDMS}$ nanocomposite strain sensors, showing its great capability in the application of detecting a small strain range with high sensitivity, which will be discussed in Section 3.

2.3 Characterization

Surface morphology and energy dispersive X-ray spectroscopy (EDX) spatial mapping of nanocomposites were characterised by a scanning electron microscope (FEI Nova SEM 450, USA). To reveal the mechanism occurred in $\text{TiO}_2@\text{CF}/\text{PDMS}$ composites, *in situ* tension tests were performed using a mechanical loading stage inside a scanning electron microscope (FEI Nova SEM230, USA). Both quasi-static and repeated cyclic loadings were measured to evaluate the piezoresistive performance of the sensors via a tensile testing machine (Instron Model 3369, USA) under displacement control mode. The same strain rate of $1.25\% \text{ s}^{-1}$ was employed for all quasi-static tension and cyclic loading. Meanwhile, a variety of strain rate within a range of 0 to $5\% \text{ s}^{-1}$ were used to study the strain rate influence of quasi-static tension test. The electrical measurement was performed at the same time with mechanical

measurement to *in situ* record the change of resistance for a sensor via a precision LCR meter (E4980AL, Keysight Technologies, USA). The response time and relaxation time measurements were proceeded by stretching the sensors at a very high strain rate of $40\% \text{ s}^{-1}$, followed by a dwelling time of 30 s and then unloading at the same strain rate.

3 Results and discussion

Figure 2 shows the surface morphology and EDX spatial mapping of TiO_2 nanowires grown on the surface of CF. TiO_2 nanowires represent a very high uniformity with the average size of 165 nm in diameter and $1.5 \mu\text{m}$ in length. Moreover, TiO_2 nanowires show a very compact growth mode. Both the first and second characterizations of $\text{TiO}_2@\text{CF}$ show a well consistent result, demonstrating the high-quality growth of TiO_2 nanowires. This can be verified by the uniform elements of Ti and O evenly distributing around the CF, as shown in Figs. 2(g) and 2(h).

Figures 3(a)–3(c) show the response and relaxation properties of the strain sensor based on $\text{TiO}_2@\text{CF}/\text{PDMS}$ composites. The response time was measured to be about 0.3701 s, including the time required for the strain sensor ramping to 5% strain (0.125 s). The response time is determined by the length of time occupied from the starting point to ending point when reacting to a given stimulus, as shown in Fig. 3. This shows a very rapid response behavior for the strain sensors based on

Fig. 1 Schematic illustration of preparation process of $\text{TiO}_2@\text{CF}/\text{PDMS}$ nanocomposites.

Fig. 2 Surface morphology and energy dispersive EDX of $\text{TiO}_2\text{@CF}$: (a) SEM surface morphology of $\text{TiO}_2\text{@CF}$; (b) zoom-in of (a) with detailed analysis of TiO_2 nanorods; (c) full scan of EDX for (b); (d–f) second characterization of surface morphology and EDX of $\text{TiO}_2\text{@CF}$ to confirm the highly uniform TiO_2 nanowires grown on CF surface.

Fig. 3 (a) Response and relaxation properties of strain sensor based on $\text{TiO}_2\text{@CF}$ /PDMS composites. Detailed analysis was performed on (b) response time and (c) relaxation time taken from the second cycle in (a). Curve fitting of exponential formula was conducted to determine the relaxation time.

$\text{TiO}_2\text{@CF}$ /PDMS composites. However, some level creep was observed, showing a sharp overshoot and a relatively long relaxation time of 0.1068 s based on the curve fitting of exponential Eq. (1) (Fig. 3), which can be attributed to the viscoelasticity of the PDMS. The relaxation time can be determined as follows:

$$\frac{\Delta R}{R_0} = a + (b - a)e^{-t/\tau} \quad (1)$$

where constant τ is called the relaxation time. The

parameters a , b , and t can be obtained through the curve fitting of experimental data, as shown in Fig. 3(c).

In addition, compared to those of reported results in literature, the response and relaxation properties of the strain sensor based on $\text{TiO}_2\text{@CF}$ /PDMS composites showed superior property [24–27]. Sensing properties of strain sensors prepared in this work were also compared to that of reported results in literature, as revealed in Table 1.

Table 1 Comparison of sensing properties between this work and reported results in literature

Material system	Sensitivity	Stretchability (%)	Linearity (%)	Reference
Cellulose/rGO/CNFs/PDMS	9.4	110	10	[28]
Silver plated fabric	26.1	3.4	2.5	[29]
CNTs@TPU/PDMS	0.19	100	12	[30]
MWCNT/silicone fluid/PDMS	26.8	40	17	[31]
Glass CF/thermoplastic	1.88	4.82	0.4	[32]
Ti ₃ C ₂ T _x /graphene/PDMS	190.8	52.6	47	[33]
Silver/PDMS	268.4	110	15	[34]
CarbonInk/SilverInk/mixtures	68	35	40	[35]
Ga/HbPAAmc/Dopa-Fe ³⁺ hydrogels	0.00854	100	12	[36]
PANI/BC/CH/PDMS	8	30	10	[37]
Piezotronic bipolar transistor	10,000	5	2	[38]
Conductive fluid/elastic rope/silicone elastomer	36	100	67	[39]
GO-AgNW/PVA-Ca ²⁺ strain sensor	105.3	52	10	[40]
CNTs film/PDMS	10	260	4.0	[41]
Printable graphite/plastic substrate	19.3	87	28	[42]
TiO ₂ @CF/PDMS	1000	55.2	10	This work

The working mechanism for resistive type strain sensors is based on the piezoresistive effect, where electrical resistance of strain sensors changes upon the external applied strain. Two factors affecting the resistance change shall be considered, that is, geometrical effect and intrinsic piezoresistive effect [43]. The equation for gauge factor of strain sensors to quantitatively reveal its sensitivity can be expressed as followings [44]:

$$\frac{\Delta R}{R_0} = (1 + 2\nu)\varepsilon + \frac{\Delta\rho}{\rho} \quad (2)$$

$$k = \frac{\Delta R / R_0}{\varepsilon} \quad (3)$$

where $\Delta R / R_0$ is the resistance change, ν is the Poisson ratio, ε is the applied strain, $\Delta\rho$ is the resistive change, ρ is the initial resistivity, and k is the gauge factor.

It is generally acknowledged that metal materials are substantially determined by the geometrical effect upon deformation, which is the first part of Eq. (1) without the second part, gauge factor $k = 1 + 2\nu$. For our studied composites, as the Poisson ratio of PDMS is about 0.5, thus, the first part contributing to the final gauge factor can be neglected owing to its small value. Therefore, the gauge factor of the studied system in this work is determined by the intrinsic piezoresistive effect, which is described in Eq. (2). To reveal its strain-sensing performance and durability under repeated cycling, cyclic loading and unloading at a

maximum strain of 5% was performed on the TiO₂@CF/PDMS composite strain sensor (Fig. 4). The variation in resistance change, which is the difference between the maximum resistance change $(\Delta R / R_0)_{\max}$ and minimum resistance change $(\Delta R / R_0)_{\min}$, was used here to assess the stability of this strains sensor. The results show that this newly developed TiO₂@CF/PDMS composite strain sensors exhibit a relatively stable performance with a slight drift under this long-term cyclic test. To explain the underlying mechanism in relation to this obtained ultra-high sensitivity, the micro-cracks formed, and propagated inside the strain sensors were put forward. High sensitivity induced by micron-scale cracks was demonstrated in

Fig. 4 Range of cyclic performance of TiO₂@CF/PDMS composite strain sensors.

printable sensors with a gauge factor up to 647 [45]. The controllable parallel microcracks in composite thin films were also contributed to high sensitivity [46]. Therefore, by introducing the controllable microcracks [1,6,47], it is practical to design and achieve strain sensors with high sensitivity. Therefore, it can be concluded that a rather high sensitivity can be achieved in TiO₂@CF/PDMS composite strain sensors.

To reveal the piezoresistive mechanism, *in situ* tension tests were performed on the fabricated TiO₂@CF/PDMS composite strain sensors. *In situ* SEM images for TiO₂@CF/PDMS composite strain sensors obtained under subsequent strains of 0%, 1%, 3%, 5%, and 10% are shown in Fig. 5. As can be seen from Figs. 5(a)–5(c), some microcracks along the direction of stretching started to emerge and the debonding phenomenon between CF and PDMS matrix gradually occurred when the strain was applied from 0% to 10%, which were indicated by the red arrows and yellow rectangular. In particular, the debonding between CF and PDMS matrix completely appeared and the CF experienced a twisting process with more cracks appearing on its surface under a 10% applied strain. When the strain was released from 10% to 0% (Figs. 5(c)–5(e)), the gap of microcracks was narrowed and the debonding level between CF and PDMS matrix was reduced. It can be concluded from Fig. 5 that TiO₂@CF/PDMS composite strain sensors undertake a maximum loading strain up to an effective range of 5%–10%. Moreover, one-dimensional TiO₂

nanowire shows the characteristic of enhanced specific surface area/aspect ratio. By growing TiO₂ nanowire at the surroundings of CF, a three-dimensional porous structure is formed, showing a tunable structural configured composite for strain sensors. This can be verified by the comparison between currently achieved results and our formerly reported results, where failure strain and sensitivity of CF/PDMS composite strain sensor show the values of 31.3%–55.5% and 20–100 (strain gauge below 10%), respectively [44]. However, in this work, the failure strain and sensitivity of TiO₂@CF/PDMS can be up to 55.2% (shown as below) and 1000, respectively, showing a pronounced improvement in both mechanical and sensing properties.

To ascertain the factors of strain rate affecting the behavior of strain sensors, stretching–holding tests with a variety of strain rates of 0.625% s^{−1}, 1.25% s^{−1}, 2.50% s^{−1}, and 5.0% s^{−1} were performed on TiO₂@CF/PDMS composite strain sensors. Figure 6(a) shows the fixed maximum strain of 5% versus time at different strain rates. Figure 6(b) shows the resistance change as a function of time at different strain rates, which is corresponding to the plots of strain versus time in Fig. 6(a). To reveal the effect of strain rate on the gauge factor of this TiO₂@CF/PDMS composite strain sensor, the calculated dynamic gauge factor based on Eq. (3) is shown in Fig. 6(c), showing the variation of gauge factor as a function of applied strain at different strain rates. When the applied strain rate was over 0.625% s^{−1},

Fig. 5 *In situ* tension test of SEM characterization in TiO₂@CF/PDMS composites.

Fig. 6 (a) Strain versus time at different strain rates; (b) resistance variation as a function of time under varied strain rates; (c) gauge factor as a function of applied strain at different strain rates; and (d) a 3D plot of resistance change, time, and applied strain at different strain rates.

Table 2 Analysis of strain-rate dependent behavior in various composite strain sensors

Material system	Strain rate	Gauge factor	Linearity	Hysteresis	Reference
GO–AgNW–C60	Increasing (0.2–0.8 mm·s ⁻¹)	No significant effect	No significant effect	Smaller	[48]
Graphene/TPU	Increasing (0.1–0.5 min ⁻¹)	Substantially increased	No significant effect	NA	[49]
Ferroelectric P(VDF–TrFE)	Increasing (10–500 mm·s ⁻¹)	Significantly increased voltage generation	NA	NA	[50]
SWNT/MWNT/TPU composites	Increasing (5–25 mm·min ⁻¹)	Weakly affected	Weakly affected	NA	[51]
Graphene based natural rubber composites	Increasing (4.5, 9.0, 18 mm·min ⁻¹)	Substantially increased (6.8, 22.9, 57.5)	Weakly affected	Increased	[52]
GNPs based multiscale composites	Increasing (2–10 mm·min ⁻¹)	Weakly affected	Weakly affected	Weakly affected	[53]
Graphene polyimide nanocomposites	Increasing (10%–1000% min ⁻¹)	Substantially increased	Weakly affected	Substantially increased	[54]
rGO/CNFs/PDMS nanocomposites	Increasing (0.005–0.1 s ⁻¹)	Weakly affected	NA	NA	[28]
CNFs/SCFs/PDMS composites	Increasing (0.0002–0.1 s ⁻¹)	Substantially increased	Weakly affected	NA	[44]
Carbon sponge/PDMS composites	Frequency (0.01–10 Hz)	Substantially increased	NA	NA	[55]
Carbon paper/PDMS composites	Frequency (0.01–10 Hz)	Substantially increased	NA	NA	[56]

such as 1.25% s⁻¹, 2.50% s⁻¹, and 5.0% s⁻¹, a peak value of gauge factor at each strain rate can be observed (Fig. 6(c)), demonstrating the significant dependence of strain rate behavior. Moreover, a rather high peak value was demonstrated in both strain rates of 1.25% s⁻¹ and 2.50% s⁻¹. Therefore, gauge factor calculated in

TiO₂@CF/PDMS composite strain sensors showed strong dependence on strain rate. This was also verified by a three-dimensional plot of resistance change, time, and applied strain in Fig. 6(d) at different strain rates, showing a large difference of resistance change at varied strain rates when performing a monotonic test. To further assure

the mechanism of the strain-rate dependent behavior in our fabricated strain sensors, we made a thoroughly analysis by comparing our results with recently reported results in literature. As shown in Table 2, a variety of fillers combined with varied polymers as matrix were employed to prepare composite strain sensors. When switching the strain rate from low to high value, PDMS polymer matrix based composite strain sensors showed a strong strain-rate dependent behavior, owing to its high viscosity property, while other strain sensors with thermoplastic polyurethanes (TPU) or natural rubbers as polymer matrix indicate a weak dependence on strain rate. Therefore, the polymer matrix plays a predominant role in the occurrence of strain-rate dependent effect in strain sensors.

To demonstrate the strain sensing performance of $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensors as wearable electronics, a sensor was subject to cyclic stretching and releasing with different roughly calculated maximum strains of 3.75%, 5%, and 10% by using the home-made designed linear stage (Fig. 7). After cyclic stretching and releasing, good durability was indicated although a slight increase of resistance change was observed owing to the unrecovered network damage formed during stretching, showing the successful achievement in designing $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensors. Therefore, the results confirm that strain sensors made of $\text{TiO}_2\text{@CF/PDMS}$ composites are very promising for detecting small strain gauge as wearable electronic devices.

4 Conclusions

TiO_2 nanowires are uniformly deposited on the

surroundings of the CF to achieve a hybrid porous structure via hydrothermal technique. By embedding into the PDMS polymer matrix, flexible $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensors are developed to meet the practical application as wearable electronics. A very rapid response of $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensors is obtained with the measured response time about 0.3701 s, including the time required for the strain sensor ramping to 5% strain (0.125 s). *In situ* tension tests are performed on the $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensors to reveal the mechanism of strain sensors under deformation, showing the debonding behavior between CF and PDMS matrix at a gradually increasingly applied strain up to 10%. Strong dependence of strain rate is revealed when performing monotonic test of $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensors at varied strain rates. Moreover, a rather high gauge factor is demonstrated in both strain rates of $1.25\% \text{ s}^{-1}$ and $2.50\% \text{ s}^{-1}$. This can be attributed to the predominant role of polymer matrix for its high viscosity property, especially for the employed PDMS as polymer matrix. Therefore, the prepared $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensors demonstrate its great potential in the application of flexible electronics at a relatively small strain gauge.

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Fig. 7 Cyclic stretching and releasing of a $\text{TiO}_2\text{@CF/PDMS}$ composite strain sensor under various of maximum applied strains: (a) cyclic performance under varied maximum strain levels and (b) distribution of relative resistance change under different maximum strain levels by colormapped line.

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