

Chapter 9

Flexible Carbon-Based Nanocomposites



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Abstract The demand for stretchable strain sensors is increasing due to their potential applications in emerging fields such as human motion detection, wearable electronics, and electronic skins. This review paper provides an overview of the latest developments and advanced applications of flexible carbonous conductive polymer composite strain sensors. Various sensing mechanisms for strain sensors, including the tunneling effect, the disconnection mechanism, and the cracking effect, are described and analyzed. Additionally, the differences in fabrication methods and sensing performance of different sensors are compared from the perspective of different morphological structures of conductive polymer composite strain sensors. The applications of flexible carbon-based strain sensors in detecting motion signals, vital signs, and other areas, such as elbow and knee flexion, gesture recognition, voice recognition, pulse, respiration, and human–computer interaction, are also discussed. Finally, the paper summarizes the current challenges that need to be overcome in the practical application of flexible conductive polymer composite strain sensors.

9.1 Introduction

The advancement of artificial intelligence and electronic technology has led to an increasing use of flexible sensors in various aspects of our lives (Lee et al. 2019). Wearable devices based on flexible sensors, including flexible strain sensors, electrochemical sensors, humidity sensors, and temperature sensors, have been developed rapidly (Chen et al. 2021a, b; Cui et al. 2019; Delipinar et al. 2021; Li and Bo 2022;

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Thakur et al. 2022; Wu et al. 2020; Zhao et al. 2022). Among these sensors, flexible strain sensors are rapidly developing with outstanding characteristics for full-range strain monitoring. Flexible and wearable electronic devices are designed to detect and quantify external signals and respond accordingly in a smart and practical manner. The recent rapid development of flexible wearable technology has provided wearability, comfort, teleoperability, and real-time response to many devices. The key component of these wearable electronics is the sensor, which converts various physiological signals, such as subtle movements caused by breathing, vocalization, and heartbeat, and large body movements caused by joint flexion, into quantifiable electrical signals (Wang et al. 2017). Furthermore, their non-invasive and real-time monitoring of vital signs provides important clinical information for preventing and rehabilitating relevant diseases. Additionally, they demonstrate great potential for human–computer interaction and robot control.

Flexible strain sensors can be fabricated using various materials, such as metal, inorganic semiconductors, and conductive polymer composites (CPCs). However, metal and inorganic semiconductor strain sensors have limitations in terms of their detection range, complex processing, and high manufacturing costs (Rahimi et al. 2015; Zhao et al. 2018; Kirubasankar et al. 2018; Lou et al. 2017). In contrast, CPCs, which consist of conductive particles in an insulating polymer matrix, have become a popular research topic due to their easy processing, adjustable electrical properties, and wide range of applications. The electrical conductivity of the polymer is determined by the conductive network created by the conductive filler (Chen, Zhu, and Jiang 2020b). Research has shown that the resistance of conductive polymers can vary with changes in the conductive network within the polymer matrix, making them suitable for use as multifunctional sensors that can monitor external stimuli such as strain and temperature (Li et al. 2020a, b, c; Li et al. 2019; Chen et al. 2020c, a, b; Arman Kuzubasoglu and Kursun Bahadir 2020; Chen et al. 2021a, b; Yu et al. 2020).

CPC-based strain sensors are ideal because of their unique combination of properties. The low-density and flexible polymer matrix allows for superior flexibility and stretchability, while the conductive fillers provide high thermal and conductive conductivity, magnetic properties, and excellent mechanical properties. The resulting strain sensors are skin-attachable, wearable, and can be arbitrarily deformed, making them superior to conventional sensors (Li et al. 2018a, b; Tang et al. 2018; Park et al. 2021; Bu et al. 2021). The wide range of potential applications for flexible strain sensors includes human motion detection, vital sign monitoring, and human–computer interaction. These sensors can detect subtle movements caused by breathing, vocalization, and heartbeat, as well as large body movements caused by joint flexion. In addition, they can provide important clinical information for the prevention and rehabilitation of relevant diseases (Kim et al. 2021; Li et al. 2021a, b, c; Wang et al. 2021a, b, c, d, e, f, g, h). Despite the advantages of CPC-based strain sensors, there are still challenges that need to be addressed before they can be practically applied. These challenges include improving the sensitivity and repeatability of the sensors, increasing their durability, and optimizing their performance

under various environmental conditions. Continued research in this field is needed to overcome these challenges and fully realize the potential of flexible strain sensors.

Appropriate material selection and preparation methods are essential to obtain flexible strain sensors with optimum performance. Currently, conductor materials used to fabricate flexible strain sensors are mainly classified into carbon materials (Xiao et al. 2021; Wang et al. 2021a, b, c, d, e, f, g, h; Feng et al. 2021), conductive polymers (Luo et al. 2022; Liu et al. 2021a, b; Wu et al. 2021), and metal nanoparticles (Liu et al. 2021a, b; Sun et al. 2022; Zhang et al. 2021a, b). Among them, carbon-based materials, including carbon nanotubes (CNTs) (Qaiser et al. 2021; Wang et al. 2021a, b, c, d, e, f, g, h), graphene (Wang et al. 2021a, b, c, d, e, f, g, h; Niu et al. 2021), carbon nanofiber (Peng et al. 2021), and carbon black (Hu et al. 2021a, b), have shown prominent prospects for development. In terms of the choice of flexible substrates, elastomers with high elongation and good flexibility, including rubber (Liu et al. 2019; Lam et al. 2021), thermoplastic polyurethane (TPU) (Wang et al. 2021a, b, c, d, e, f, g, h; Tang et al. 2022), polydimethylsiloxane (PDMS) (Soe et al. 2021; Zhang et al. 2021a, b), and polyimide (PI) (Li et al. 2021a, b, c; Pu et al. 2021), have been extensively reported in the literatures. A stable interaction between the carbon filler and the elastomeric matrix is essential for the sensing performance of the CPCs. Typically, CPC is obtained by incorporating a conductive filler with an insulating polymer matrix using suitable processing techniques such as spin-coating (Soe et al. 2021), spray-coating (Hu et al. 2021a, b; Hu et al. 2021a, b) and solution mixing (Zheng et al. 2021). Continuous conductive networks are established based on interconnections of neighboring conductive fillers as the filler loading increases. The transition of the resulting material from insulator to conductor can occur at a threshold content of filler, which is known as the percolation threshold (P_c). The lesser amount of filler, i.e. lower P_c , can maintain the good electrical properties of CPC while preserving the inherent processability and mechanical properties of the polymer (Mei et al. 2021; Lv et al. 2021). The percolation threshold is typically determined experimentally by measuring the electrical conductivity of a composite material as a function of the concentration of the conductive filler. A common method is to prepare a series of samples with different filler loadings and measure their conductivity using a four-point probe or other suitable method. The percolation threshold is then identified as the concentration at which the conductivity starts to increase rapidly.

In recent years, there have been numerous studies on the development of methods for combining carbon-based materials with polymers to improve the strain-sensing properties of the resulting composites. These methods can be broadly classified into two categories based on the distribution morphology of the carbon-based materials: homogeneous blends and ordered arrangements. Homogeneous blends refer to the uniform dispersion of materials throughout the polymer matrix, while ordered arrangements involve the controlled alignment of materials within the polymer matrix. Different assembly methods endowed the conductive polymer composites with different structures, including membranes Li et al. 2021a, b, c; Yang et al. 2021), fibers (Sheng et al. 2021; Zeng et al. 2021a), yarns (Tang et al. 2022; Jang et al. 2021; Zhao and Xu 2022), sponges (Xu et al. 2021; Bai et al. 2022; Huang et al.

2021), and foams (Wang et al. 2021a, b, c, d, e, f, g, h; Veeralingam et al. 2022), which had an impact on the resulting strain sensor performance.

This paper aims to provide an in-depth and comprehensive review of recent advances in carbon-based CPC flexible strain sensors. This review covers a wide range of topics, including the sensing mechanisms of strain sensors, the use of various carbon conductive materials in strain sensors, the impact of different sensor structures and assembly methods on sensing performance, and the latest applications of flexible strain sensors for signal monitoring. Furthermore, the paper not only summarizes the current state of the art in flexible strain sensors but also predicts future trends in this field. Through presenting the latest research on carbon-based flexible strain sensors and offering a comprehensive reference, the main aim of this paper is to facilitate a more profound and extensive understanding of this promising and increasingly popular research area.

9.2 Strain Sensing Mechanism

The sensing mechanism of strain sensors is the fundamental principle underlying the detection of strain-induced changes in electrical conductivity within the CPC material. To accurately measure the degree of strain, it is necessary to adjust the conductive network within the material in a manner that is both responsive and reliable. This section will comprehensively discuss various mechanisms that describe the strain sensing behavior of CPC strain sensors, including the tunneling effect, disconnection of a coupled conductive network, and crack extension mechanisms.

9.2.1 Tunneling Effect

In general, the conductivity of CPC is determined by the formation of a conductive network from the conductive filler. The formation of a conductive network can be divided into two main types, one is the tunneling effect and the other is the physical contact of the conductive filler. Tunneling effect, also known as quantum tunneling, refers to the phenomenon in which particles can pass through a barrier that, according to classical physics, they should not be able to overcome. This occurs because of the wave-like nature of particles at the quantum level. In classical mechanics, if a particle encounters a barrier that is higher than its kinetic energy, it will bounce off the barrier and be unable to penetrate it. However, in quantum mechanics, particles have a wave-like nature and are described by a probability wave function. This means that a particle can actually pass through a barrier that it doesn't have enough energy to surmount, by tunneling through the barrier (Lantada et al. 2010; Hu et al. 2008).

Tunneling effect provides the possibility for insulating matrix with low filler content to be transformed into a conductor. In the electrical behavior of CPCs, the tunneling effect mainly exists in CPCs with low filler content. Typically, there are

insulating polymer barriers between the fillers in these CPCs, which are located above the level of free electrons and can prevent the flow of electrical current. When the filler particles are sufficiently separated from each other, there is usually no electrical current in the CPCs. However, when the distance between the filler particles is small enough, tunneling current may occur. The total resistance R of the CPC is calculated according to the available models as follows (Chen et al. 2007; Zhang et al. 2000),

$$R = \left(\frac{L}{N}\right) \left(\frac{8hs}{3a^2e^2\gamma}\right) \exp(\gamma s) \quad (9.1)$$

where L is the number of filler particles forming a single conducting path, N is the number of conducting paths, h is Planck constant, s is the distance between filler particles, a^2 is the effective cross-section at which tunneling occurs, e is the electron charge, and γ is calculated as follows,

$$\gamma = \frac{4\pi}{h} \sqrt{2m\varphi} \quad (9.2)$$

where m is the mass of the electron, φ is the height of the potential barrier between adjacent particles.

It is evident that R is proportional to s , making s a crucial parameter that influences the tunneling effect between adjacent conductive fillers. It is widely accepted that s decreases with the increasing quantity of conductive fillers, resulting in a lower R of the CPC. As the filler content increases further, until the Pc is reached, the interconnected fillers will establish an even lower resistance conductive network within the polymer matrix. At this point, the interconnected conductive network gradually replaces the tunneling effect as the main conducting mechanism.

In the case of a CPC under stress, the resistance changes as the distance of the filler particles changes. Assuming that the distance between the filler particles is s_0 in the initial state, changes to s after the applied strain and the number of conductive paths changes from N_0 to N , the relative resistance (R/R_0) can be calculated from Eqs. (9.3), (9.4) and (9.5) (Chen et al. 2019).

$$\frac{R}{R_0} = \left(\frac{Ns}{N_0s_0}\right) \exp[\gamma(s - s_0)] \quad (9.3)$$

$$s = s_0(1 + C\varepsilon) = s_0 \left[1 + C \left(\frac{\Delta l}{l_0} \right) \right] \quad (9.4)$$

$$N = \frac{N_0}{\exp(M\varepsilon + W\varepsilon^2 + U^3 + V^4)} \quad (9.5)$$

where R_0 and s_0 are the initial resistance and the initial particle distance, ε is the tensile strain of the CPC, Δl and l_0 refer to the deformation and the initial length of the CPC, respectively. M , W , U , and V are constants.

This mechanism is suitable for relatively small strain amplitudes where a varying tunneling distance results in a change in resistance and thus a small induced signal. Some literature has validated the accuracy of the tunnel conduction model from experimental test data on different types of CPC strain sensors (Ji et al. 2014; Luo et al. 2018; Lin et al. 2013).

9.2.2 Network Disconnection Mechanism

As mentioned above, with an increase in filler content, the conductive mechanism of CPC changes from a tunneling effect to the formation of an interconnected conductive network, i.e. the percolation theory. According to the classical statistical percolation model, when the filler loading exceeds P_c , the relationship between the filler loading and the conductivity of the CPC can be expressed as (Bershtein et al. 2002),

$$\sigma = \sigma_0(p - p_c)^t \quad (9.6)$$

where σ is the conductivity of the CPC, σ_0 is the conductivity of conductive fillers, p is the volume fraction of the conductive filler in the CPC, p_c is the percolation threshold, and t is the exponent.

In general, two-dimensional conductive networks can be realized with a t of 1.1–1.3, and three-dimensional (3D) conductive networks are considered possible when the t is in the range 1.6–2.0.

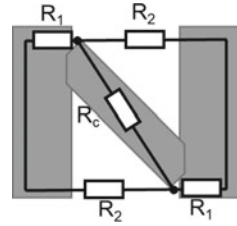
When the CPC is under strain, the large stiffness mismatch between the conductive fillers and the flexible polymer due to weak interfacial bonding and the conductive nanomaterial slips, leads to an overlap disconnection and consequently an increase in resistance. The strain-sensing mechanism of CPCs constructed from flexible polymers and multidimensional conductive fillers is commonly explained by a disconnection mechanism (Alamusi et al. 2011; Desai and Haque 2005; Li et al. 2018a, b).

On removal of the external strain, the conductive network within the CPC returns to its initial state in conjunction with the polymer matrix. However, the hysteresis effect of the polymer molecular chains and the irrecoverable damage to a few of the conductive paths result in the resistance of the CPC not being recovered to its initial value. This phenomenon above the initial resistance is known as residual resistance and is apparently one of the factors affecting the stability of the CPC strain sensing performance.

9.2.3 Network Crack Mechanism

It is widely recognized that tunneling conduction and disconnection mechanisms can be used to describe the strain-sensing behavior of homogeneous CPC strain

Fig. 9.1 The resistance model of a sensing unit



sensors. Recently, a new sensing mechanism, called network crack mechanism, has been discovered by researchers studying the strain sensing properties of sandwich structure CPC (Wang et al. 2018a, b).

The sandwich structure CPC consists of at least one conductive layer and one high elasticity polymer layer. Due to the difference in stiffness between the two layers, when a strain is applied to the CPC, the conductive layer is not able to sustain the strain and produces cracks perpendicular to the direction of stretch. Within a limited range of strain stretching, such cracks are evenly distributed in the conductive layer, rather than penetrating the entire material leading to the fracture of the material and the conductive network. There is no doubt that the formation of cracks destroys part of the conductive network and leads to an increase in the resistance of the CPC. After strain removal, the crack will re-close as the elastic layer recovers its initial state. Such reproducible disconnections and connections of the cracks enable a large sensing range and excellent repeatability. Several scholars have already designed strain sensors with a high detection range based on this mechanism (Yang et al. 2018; Wang et al. 2014).

It has been shown that most cracks will break at 60% strain, but bridges between adjacent cracks can effectively prevent the conductive network from rupturing monolithically. A simplified resistance model based on the network crack has also been proposed (Wang et al. 2018a, b), as shown in Fig. 9.1.

The resistance can be calculated by as,

$$R = \frac{R_1 R_C + 2R_1 R_2 + R_2 R_C}{R_1 + 2R_C + R_2} \tag{9.7}$$

where R_1 , R_2 , and R_C are the resistances of the island, crack gap, and bridge, respectively.

In conclusion, the sensing mechanism of strain sensors is complex and highly determined by the composition of the CPCs and the structure of the internal conductive network. Excellent strain sensing performance is often the result of a synergy of various sensing mechanisms. In general, the tunneling effect imparts sensitivity to a small range of strains, the disconnection mechanism ensures repeatability and the network crack mechanism extends the effective strain sensing range.

9.3 Carbon-Based Strain Sensors with Special Structure Design

The morphological structure of CPCs plays a critical role in their sensing performance, as it governs the distribution and connectivity of conductive fillers within the polymer matrix. To achieve the desired sensing performance, it is crucial to design and fabricate CPC sensors with specific structures that enable effective strain sensing. Currently, there is significant research focused on designing a range of sensor structures through the synergistic conformation of micro/nanostructures, which has proven to be an effective approach to creating functional CPCs. In this section, we will classify the current carbon-based CPC strain sensor structures and provide detailed discussions on the preparation methods of each structure and their effects on sensing performance, providing insights into the optimization of CPC sensor design for improved sensing performance.

9.3.1 Carbon-Based Film Strain Sensors

Thin films, due to their high sensitivity and flexibility, have gained great interest as a promising structure in the field of developing strain sensors. Thin film CPC sensors can be prepared using various methods, including wet and dry methods, which offer distinct advantages and disadvantages.

Wet methods are, by definition, the manufacture of thin films from a liquid phase dispersion of carbon material. Wet methods include solution casting, spin coating, spray coating, dip coating, vacuum filtration, compression moulding, electrophoretic deposition, and layer-by-layer self-assembly. The fabrication of CPC films by solution casting involves casting a dispersion consisting of fillers and polymers into the substrate mould, followed by drying and curing to obtain a film that adheres to the surface of the substrate. Based on this, the uniformity of the film can be further improved by using the spin coating method. In this way, the same dispersion is deposited in the center of the substrate and the dispersion is uniformly diffused by the centrifugal force of the high-speed rotation of the base. Spraying coating is the process of applying the dispersion to the substrate by using a sprayer. The involvement of the sprayer enables a thinner film to be created, also as a way of improving uniformity. Compression molding entails casting the CPC dispersion into a mold and curing it under pressure and heat within the mold. Under the influence of an electric field, the charged filler particles in a dispersed solution migrate toward the electrodes, forming a homogenous film during the electrophoretic deposition process (Fu and Yu 2014). For example, a simple and effective compression mold method, as shown in Fig. 9.2a, was reported for the preparation of natural rubber/primitive graphene film sensors. The composite had a graphene loading of 5 wt% and a conductivity of 0.005 S/m, which was significantly higher than other methods (Liu et al. 2019).

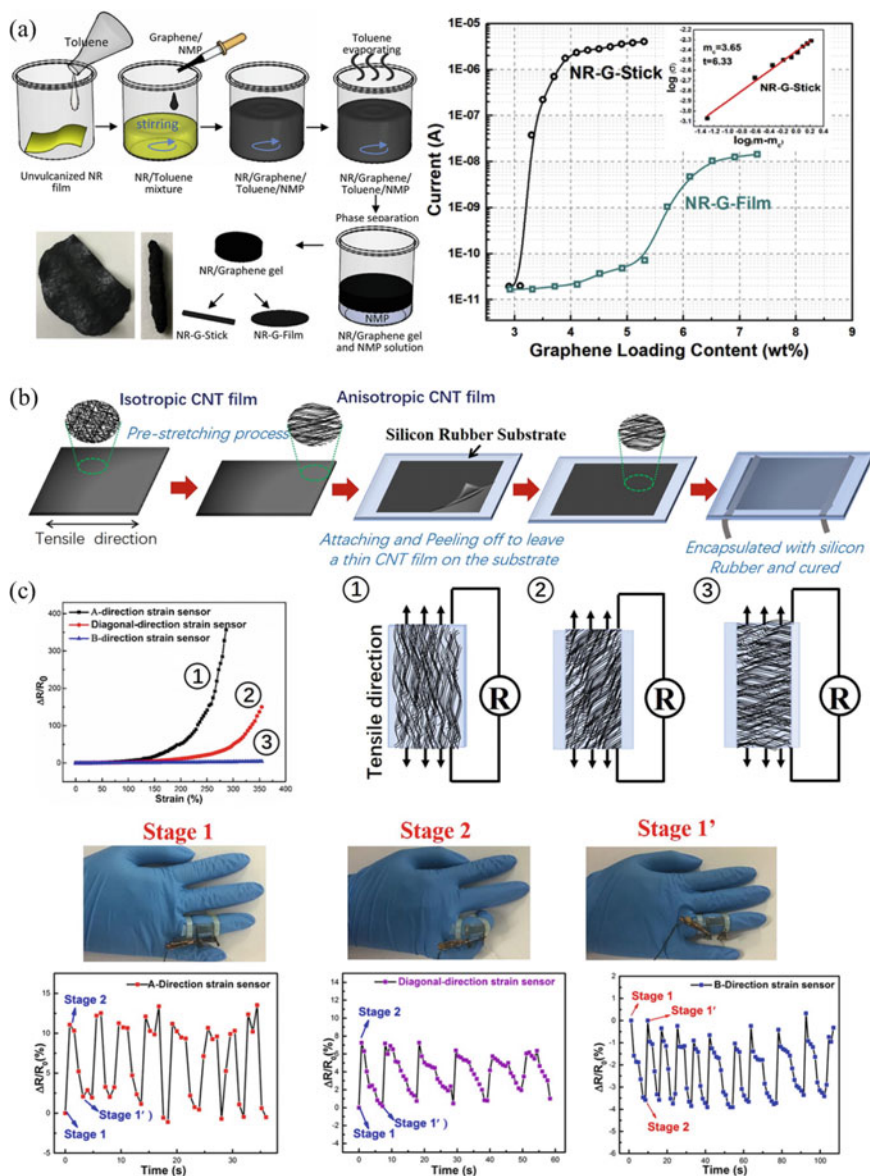


Fig. 9.2 a Preparation process and conductivity of natural rubber/primitive graphene film (Liu et al. 2019). b Schematics of the fabrication process of the aligned CNT film. c The anisotropic feature of CNT film-based strain sensors (Ma and Lu 2020) (Reprinted from references, with kind permission of Elsevier Publications)

Dry methods involve the formation of thin films directly from filler arrays including chemical vapor deposition (CVD) growth and direct dry transfer. Typically, the CVD process allows hydrocarbon gases to pass through a tubular reactor at high temperatures, where the hydrocarbon gases thermally decompose on the surface of a substrate coated with a metal catalyst to form graphene films. Direct dry transfer is the transfer of conductive fillers onto a polymeric substrate under hot pressing and mechanical peeling. Layer-by-layer self-assembly processes use electrostatic interactions, hydrogen bonding, or covalent bonding to alternately deposit oppositely charged components (polymers and fillers) on a substrate. The several layers that make up the film overlap and interpenetrate one other (Zhang et al. 2018). Numerous carbon-based film strain sensors based on these methods have been reported in the literature. The pre-stretching process has been reported to align the CNT film prepared by CVD on a silicone rubber substrate to prepare a highly sensitive and flexible bi-directional strain sensor (Fig. 9.2) (Ma and Lu 2020). The results indicate that the arrangement of CNTs in the axial direction and their entanglement in the transverse direction contribute to providing strain-sensing capabilities in different directions. The authors also highlighted the demonstration of joint motion detection as evidence of the potential of this flexible, bi-directional strain sensor in detecting complex large deformations, as shown in Fig. 9.2c. The fabrication of reduced graphene oxide-TiO₂ composite films by spraying methods is proposed to develop a multifunctional material for strain sensing and photodegradation applications. The obtained multifunctional composite films show high optical transmittance and excellent strain sensing properties with a gauge factor (GF) of 12–23 (Liu and Zhang 2016). Moreover, a similar approach was used by Yu et al. to prepare a strain sensor by direct coating of aligned CNT films on a PDMS substrate (Yu et al. 2017). The strain sensors are extremely durable, with a strain capacity of up to 400% and a response time of 98 ms, thanks to their superior mechanical qualities. However, the GF of the sensors resulting in the above work was poor, which is attributable to the thickness of the conductive film. These results show that by simply attaching fillers to the surface of an elastic substrate it is difficult to obtain superior sensors. Sandwich-structured films obtained by embedding filler films into polymers are considered to be a better way to fabricate strain sensors. Zhou et al. (2017), prepared strain sensors by embedding fragmented SWCNT paper into PDMS and demonstrated that a GF of more than 107 was guaranteed even at 50% strain. The sensors obtained by this technique possess unprecedented robustness and sensitivity.

A flexible polyurethane/graphene sheet composite film was manufactured by the layer-by-layer method for wearable strain sensors. It exhibited an excellent conductivity of 1430 ± 50 S/cm and a GF of up to 150 (Meng et al. 2019). In addition, rapid assembly of graphene films at the liquid/gas interface via the Marangoni effect has been reported (Li et al. 2016a, b). These graphene-based strain sensors exhibited extremely high sensitivity at 2% strain, with GF as high as 1037. These sensors can be utilized in electrodynamic systems to monitor large-scale bodily movements.

9.3.2 Carbon-Base Foam Strain Sensor

Porous foam structures can be designed for use in carbon-based strain sensors to detect varying degrees of compression and tension. The unique porous structure of the composite foam material gives it excellent elasticity and recoverability, a characteristic that ensures an excellent strain range and stability of the strain sensor. Strain sensors with porous foam structures are prepared by template-assisted methods, freeze-drying, and CVD. The size of these pores can be tuned by varying the template structure and the preparation process.

In the preparation of CPCs using template-assisted methods, sugar is the most commonly used template due to its low cost and ease of removal (Hwang et al. 2021; Sun et al. 2018; Wan et al. 2020; González-Rivera et al. 2018). For example, a carbon black/polydimethylsiloxane (PDMS) foam (CPF) was fabricated using a templating method combined with ultrasound technology for use as a stretchable sensor, as shown in Fig. 9.3a. The CPF, featuring a robust 3D conductive network, exhibits excellent linearity and a wide strain range. Owing to the strong interaction between the conductive nanofillers and the PDMS matrix, the fabricated CPF possesses negligible electrical hysteresis (Xia et al. 2021). Iglío et al. (2018), reported a piezoresistive flexible foam sensor that can simultaneously detect ultralow strain less than 0.1% and pressure of 20 Pa in compression mode. This PDMS/CNT foam sensor was prepared using a low-cost and scalable process by the molding of a sugar template and subsequent drop-casting of CNT ink. The good stability and limited hysteresis were evident by performing 255 compression release cycles at different strain rates of up to 10 mm/min over a strain range of 0–60%. The same method was used to prepare a 3D microporous graphene-coated PDMS sponge for highly elastic and wearable piezoresistive strain sensors suitable for attachment to human skin (Jung et al. 2019). The obtained graphene-coated PDMS sponges showed highly stable mechanical properties in various tensile stress–strain tests. When various strains were applied, the resistance of the device varied in a highly stable, repeatable, and reversible manner. Besides, the use of other chemicals as pore-forming agents is also a simple and effective way to obtain porous CPC strain sensors. For instance, Long et al. (2018), reported a chemical foaming method using ammonium bicarbonate powder to prepare porous CPCs using PDMS as a matrix and graphene as a conductive filler for the detection of tensile and compressive strains.

Freeze-drying can also be used to prepare three-dimensional carbon foams. By using the freeze-drying process, porous CNT/TPU and graphene/TPU nanocomposites were prepared by Liu's group (Liu et al. 2018). The density of the two carbon-based TPU conductive foams was only 0.1 g/cm³, and the compression rate exceeded 90%. The resistance of the porous CNT/TPU showed a decreasing trend during compression due to the densification of the porous structure and the opposite piezoresistive behavior was observed due to the disruption of the defective cell walls. After cyclic load stabilization, both porous foams showed excellent recoverability and reproducibility when cyclically compressed at wide strains of up to 90%. Similarly, Li et al. (2016a, b) provided a self-assembly method to form graphene hydrogels

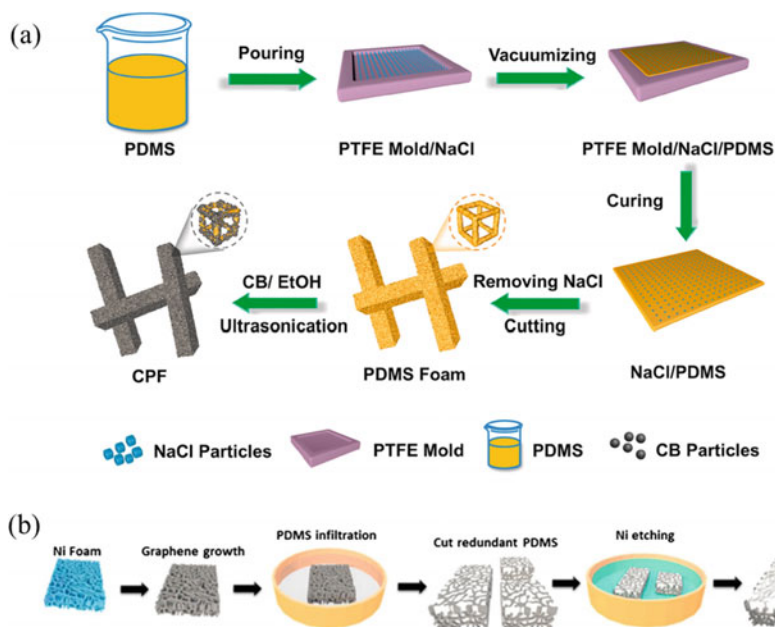


Fig. 9.3 **a** Fabrication of carbon black/PDMS foam through a sacrificial template method (Xia et al. 2021). **b** Fabrication process of graphene foam/PDMS sensor (Pang et al. 2016) (Reprinted from references, with kind permission of Elsevier and ACS Publications)

and obtained graphene foams by a freeze-drying process. Due to its friability, the foam was inserted into a PDMS to be used as a strain sensor. The sensors obtained by embedding the foam into PDMS elastomers showed a strain range of more than 20% and the relative resistance change showed a high degree of superposition with the strain curve during the stretch-relaxation cycle.

Graphene foams are prepared by the CVD method, typically using nickel and copper foams as templates (Ma et al. 2017). In the preparation process, graphene oxide was first grown on the metal foam by the CVD method, followed by the reduction of the graphene oxide to graphene. Finally, the metal skeleton was removed by etching to retain the porous structure of the template. Graphene foam has ultralightweight properties. This material needs to be combined with an elastic polymer in order to form a stable structure during compressive strain. As a compressive strain sensor, the higher the raw resistance of the conductive material, the higher the sensitivity. A graphene foam/PDMS/PET CPC strain sensor was also reported, which was fabricated by infiltrating PDMS into 3D graphene foam and introducing a thin layer of PET as a substrate (Xu et al. 2014). However, the performance did not show advantages in terms of sensing performance, with a maximum strain of approximately 16% and a GF of only 6.24. A strain sensor combining graphene porous foam with PDMS was also demonstrated by Pang et al. (2016), as shown in Fig. 9.3b. Using nickel foam as a template and a chemical etching method, graphene foam sensors

can be prepared in PDMS-nickel foams coated with graphene, which can achieve both pressure and strain-sensing properties. Graphene foam-based sensors exhibited a wide pressure sensing range and highest sensitivity as pressure and strain sensors respectively. Generally, the strain range of carbon-based foams was lower than that of carbon-based films, typically below 50%, and there was no significant advantage in sensing sensitivity. The primary reason for such results is the fact that the conductive network within the foam structure is more difficult to vary during the deformation process (Yan et al. 2018).

9.3.3 Carbon-Base Textile Strain Sensor

Textiles, due to their naturally flexible structure and wearability, are more suitable for use as strain sensors to monitor the physiological signals emitted by the human body. A great deal of work has been done to develop textile-based sensors and apply them to monitor the human body in real-time and to detect various other signals (Li et al. 2020a, b, c; Lu et al. 2019; Yue et al. 2020; Sun et al. 2019; Gao et al. 2020; Wang et al. 2020; Zheng et al. 2020; Feng et al. 2020). Textiles are usually divided into three categories: fibers, yarns, and fabrics. Fibers are the most basic unit form that constitutes a textile and have a very high length-to-diameter ratio. The twisting of fiber bundles results in yarns, while further techniques such as weaving and knitting produce fabrics. In this section, carbon-based strain sensors based on textile structures realized using various manufacturing methods are presented.

The slender morphological structure of fibers gives them a natural advantage for use in detecting tensile deformation. As biomass fibers, natural silk, and hair fibers can be used alone as strain-sensing substrates due to their sufficient length and natural elasticity. The active groups on their surfaces facilitate the bonding to carbon-based materials. By incorporating ultra-fine graphite flakes on the surface of the silk fibers, researchers have developed a strain sensor with a GF of 14.5 and a strain range of 15% (Zhang et al. 2016). The flexible and elastic characteristics of the silk fibers guaranteed that the sensor could maintain its performance after 3000 cycles of loading and releasing. Yuan et al. (2015) have developed strain sensors based on rGO-coated hairs. These sensors can detect tensile, bending, and compression deformations. They have excellent mechanical stability and repeatability, with moderate sensitivity. The excellent mechanical properties of chemical fibers, including high stress and elasticity, enable them to be used as a base material for carbon-based strain sensors. Wang et al. (2018a, b) reported the preparation of TPU and MWCNT-based fibrous strain sensors by wet-spinning method. In tensile tests, the MWCNTs/TPU fibrous sensors showed an effective strain range of up to 320% and a strain coefficient of 97.1 in the high tensile range. The durability of the MWCNTs/TPU fibrous sensors was also examined by means of a 100% tensile cycle test at 9700 cycles. In the same way, Zeng et al. (2021b) developed polystyrene-block-poly (ethylene butylene)-block polystyrene (SEBS)/CNT hybrid fibers with high electrical conductivity, high strength, and elongation at break. The hybrid fiber-based strain sensors

have an ultra-wide sensing range of 471%, a high GF of 368, and excellent reliability and stability. To overcome the challenges of the adhesive properties of elastic polymers, researchers have developed a novel self-healing carbon nanotube/ethylene vinyl acetate (CNTs/EVA) fiber strain sensor by embedding CNTs into the surface of an expandable shape memory EVA fiber using ultrasound. The CNTs/EVA fiber strain sensor exhibits significant response capabilities, including high stretchability, a large linear working range, excellent dynamic durability, and a fast response time. Furthermore, the mechanism of CNTs/EVA fiber conductive network changes at the stretched state and unstretched state was discussed, as shown in Fig. 9.4a (Li et al. 2020a, b, c). The strain sensors obtained using this hybrid fiber can accurately detect subtle muscular and large-scale body movements due to their excellent performance. Meanwhile, the fibers traditionally have a feature for use in clothing, satisfying their use in garments and other flexible electronic devices to monitor human movement signals.

Compared to fibers, yarns are structurally more stable due to the twisting effect and are ideal for strain sensors. Wang et al. (2016a, b) have produced a composite yarn for strain sensors by coating highly conductive SWCNT with an elastic cotton/PU core yarn. Thanks to the cotton/PU yarn covering structure and the reinforcing effect of the SWCNTs, the composite yarn can withstand strains of up to 300% and can be cycled nearly 300,000 times at 40% strain without significant breakage. A CNT and PU nanofiber composite spiral yarn with electrical conductivity, hyperextension, and high tensile sensitivity has been developed (Gao et al. 2020). The spring-like microscopic geometry and the good elasticity of the PU molecules give the spiral yarn excellent stretching properties (Fig. 9.4b). CNTs/PU spiral yarns achieve good recovery up to 900% and maximum tensile elongation of up to 1700% due to the interlaced conductive network at the micro level and the spiral structure at the macro level. In addition, Park et al. (2015) developed various yarn strain sensors by dip-coating polyvinyl alcohol and graphene nanosheets layer by layer onto rubber, nylon-covered rubber, and wool yarns, respectively. Due to the yarn structure, these sensors were highly stretchable (up to 150%) and versatile, and capable of detecting human motion on both large and small scales.

Fabrics obtained by weaving or knitting yarns allow further integration of strain sensors into wearable devices. In other words, fabric sensors can be worn without any supporting substrate or additional accessories. Different fabric tissues also provide adjustability in the sensing range, sensitivity, and stability of the fabric sensor. The early fabric strain sensors were fabricated from tubular plain knitted fabrics woven from steel or carbon fiber yarns (Park et al. 2015). Although the resulting fabrics were highly conductive, the strain-sensing range was only 20%. Ren et al. (2017) prepared graphene/cotton fabrics by depositing graphene oxide onto cotton fabrics by vacuum filtration and then hot pressing the material to reduce the graphene oxide. However, due to its compact structure, the graphene layer could not stretch large strains. In order to increase the sensing range, a study was carried out to create a fabric strain sensor by continuously winding CNT around a spandex yarn during the knitting process (Foroughi et al. 2016). The developed fabric showed a strain-sensing range of up to 100%. After 1000 stretch-release cycles at 100% strain, the

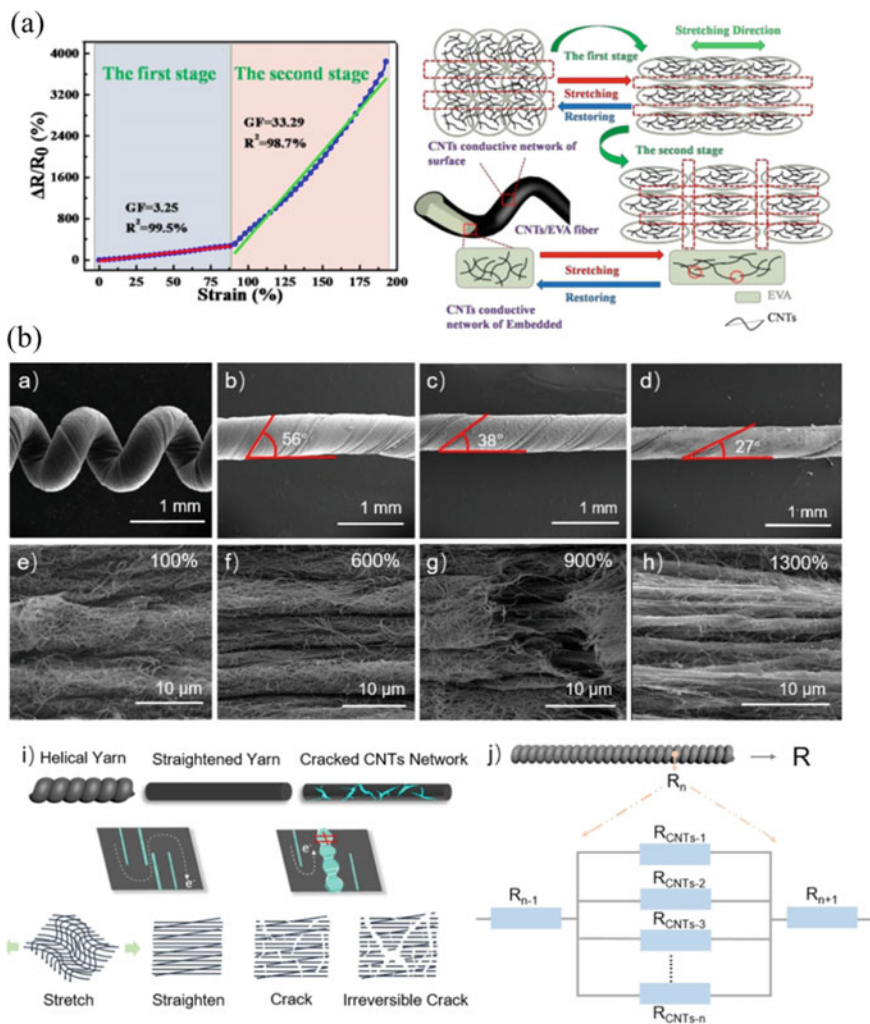


Fig. 9.4 **a** The relative resistance change of CNTs/EVA fiber strain sensors and the mechanism of CNTs/EVA fiber conductive network changes at different states (Li et al. 2020a, b, c). **b** SEM images and mechanism illustration of the conductive network of the helical CNTs/PU yarn (Gao et al. 2020) (Reprinted from references, with kind permission of ACS Publications)

sensitivity was still largely maintained. Wang et al. (2016a, b) developed an ultra-highly stretchable and extremely sensitive strain sensor derived from a carbonized silk fabric. Thanks to the hierarchical structure of the fabric, these fabric-based strain sensors have a large sensing range of up to 500% and long-term stability. In addition, a new graphene woven fabric sensor with an ultra-high GF of approximately 103 and a detection range of 6% strain was developed by the CVD method (Wang et al. 2014). In addition to conventional textile knitted and woven structures, non-woven

fabrics can also be used as sensor substrates. For example, nonwoven sensor has been prepared by integrating graphene directly onto a nonwoven fabric and reducing it in HI acid (Du et al. 2016). The prepared sensor exhibited a negative strain coefficient at small strains and the signal was reproducible in response to tension, bending, and pressure.

In summary, woven fabric-based strain sensors offer ultra-low detection limits, high sensitivity, excellent cycling stability, and durability due to the high dimensional stability of the woven structure. Knitted fabric-based strain sensors, on the other hand, offer excellent sensing sensitivity and a customizable sensing range. Different fabric constructions offer options for sensor design and manufacture, allowing for a greater variety of strain sensor construction forms.

9.3.4 Applications of the Carbon-Based Strain Sensors

As discussed above, carbon-based CPCs were developed into flexible strain sensors with varying morphological structures and excellent performance. By attaching these carbon-based sensors directly or indirectly to the body, various signals generated by the human body can be effectively detected. This section describes the various applications of carbon-based CPC strain sensors.

9.3.5 Human Motion Detection

In general, human motion can be divided into two categories, the first being strenuous movements such as running, walking, and limb flexion, and the second being subtle movements such as, for example, blinking, speaking, and facial expressions. The performance requirements for strain sensors for detecting these two forms of motion signals are different, with the former requiring a greater operating range and the latter requiring exceptionally high sensitivity at low strain (Yin et al. 2017). For the monitoring of strenuous movements, flexible strain sensors with a wide operating range and reliability can be assembled into wearable textiles. For example, by attaching flexible CPC strain sensors to sleeves and trousers, elbow and knee flexion can be sensitively detected (Suzuki et al. 2016; Zhang et al. 2020a, b), as shown in Fig. 9.5a, b. Moreover, it is possible to ultimately recognize different gestures by attaching flexible sensors to the fingers, by outputting different signals of resistance changes, as shown in Fig. 9.5c, d (Suzuki et al. 2016; Li et al. 2020a, b, c).

For the monitoring of small movement signals, flexible strain sensors with excellent low-strain sensitivity are required. For instance, the different movement signals generated by the corners of the mouth when a person chews and speaks can be captured and analyzed by a flexible sensor attached to the cheek, as in Fig. 9.5e (Chen et al. 2020a). Similarly, the changes in throat movement produced when different words are spoken can be monitored with flexible strain sensors (Fig. 9.5f) (Xue et al.

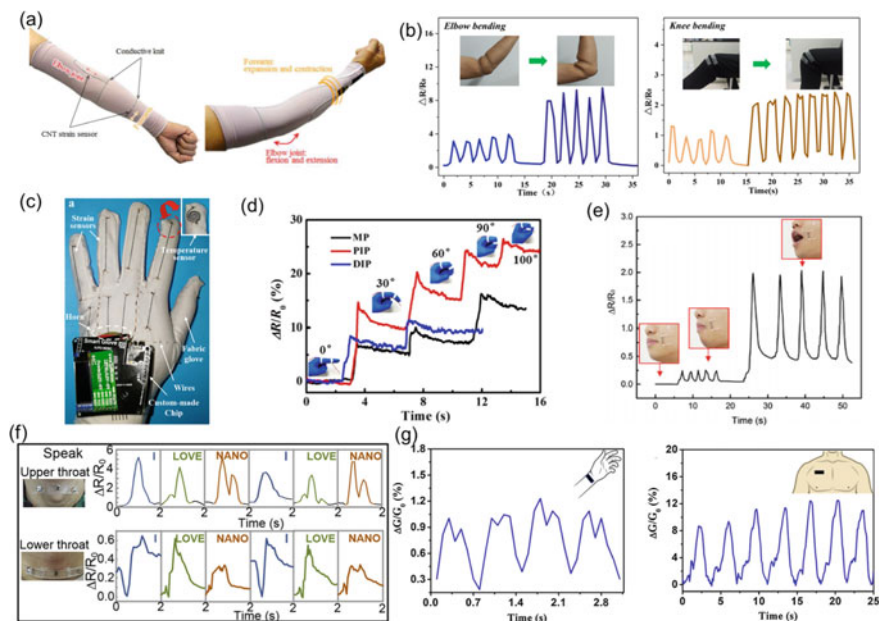


Fig. 9.5 Application of the carbon-based CPC strain sensors. **a** Sleeve with a CNT strain sensor (Suzuki et al. 2016). **b** Monitoring of human motions using flexible strain sensors: elbow bending and knee bending (Zhang et al. 2020a, b). **c** Data glove with CNT strain sensors. **d** MWNTs/PDMS sensor for finger bend detection (Li et al. 2020a, b, c). **e** Responses of the PDMS/CNTs/PDMS motions of cheek (chewing and yawning) (Chen et al. 2020a). **f** The changes in throat movement when saying different words are monitored by sensor (Xue et al. 2019). **g** Real-time human bio-signals monitoring: pulse beat and respiration (Yin et al. 2019) (Reprinted from references, with kind permission of Elsevier and ACS Publications)

2019). In addition, facial expressions such as crying, laughing, and blinking can be monitored by mounting flexible strain sensors on the cheeks, forehead, or around the eyes (Meng et al. 2019; Zhao et al. 2019).

9.3.6 Detection of the Information on Human Vital Signs

In addition to monitoring motion signals, strain sensors can also be used to detect information on human vital signs, including blood pressure, heartbeat, and respiration. Such signals are much weaker than motion and therefore smart, flexible sensors with high sensitivity to small deformations need to be prepared. Flexible carbon-based strain sensors proved to be suitable for the detection of vital signs.

Numerous reports have been made of high-performance strain sensors placed on the chest for monitoring respiratory rate, a deformation created by changes in the undulation of the chest acting on the sensor during breathing (Zhang et al. 2020a, b;

Zhao et al. 2019; Yin et al. 2019). Strain sensors can also detect pulses to assess the physiological status of the cardiovascular system for health monitoring. Similarly, by fixing a flexible carbon-based strain sensor to the wrist, the weak deformation generated by the pulse was recognized and recorded, allowing information about the heart rate to be monitored and output (Yin et al. 2019; Zu et al. 2020). Flexible strain sensors can also be implanted in the body and wrapped around blood arteries during surgery to continuously monitor vascular pressure and flow (Majerus et al. 2018). In conclusion, highly sensitive carbon-based strain sensors are making tremendous progress in the detection of physiological information in the human body.

9.3.7 Other Applications

The low strain range sensitivity of strain sensors can also make them candidates for structural damage monitoring, which can be applied to the monitoring of internal damage in structural materials. For example, PVA/CNT fibers were embedded in glass fiber-reinforced composites to enable monitoring of damage to the composite when subjected to external loads (Alexopoulos et al. 2010). Real-time monitoring has shown that micro-cracks and fractures within the composite can generate signals of changes in electrical resistance, thereby preventing further damage. Moreover, future intelligent robots will rely heavily on flexible strain sensors (Zhang et al. 2020a, b). When the strain sensing system is configured as a multi-channel structure, precise strain detection for each of the intelligent robot's fingers is possible, either concurrently or sequentially. Furthermore, highly compliant, low-modulus, and lightweight stretchable sensors can be integrated with machine learning technologies, offering unlimited prospects for bioelectrical signal recognition, tactile sensing, and multimodal integration (Wang et al. 2021a, b, c, d, e, f, g, h).

9.3.8 Conclusion and Outlook

This review presented the latest developments in flexible carbon-based CPC strain sensors and their advanced applications in various aspects. Firstly, we analyze the various sensing mechanisms of strain sensors, mainly including the tunneling effect, the disconnection mechanism, and the cracking effect. Despite the existence of different sensing mechanisms, all can be attributed to changes in resistance due to material deformation at a macroscopic level. Subsequently, the fabrication methods and sensing performance of different sensors are compared in terms of different morphological structures of CPC strain sensors. There is no doubt that the operating range and sensitivity of the sensors can be tuned by the choice of substrate material and the design of the morphological structure. Furthermore, applications of flexible carbon-based strain sensors in the detection of motion signals, vital signs, and other

areas are cited, including elbow and knee flexion, gesture recognition, voice recognition, pulse, respiration, and human–computer interaction. So far, a number of flexible CPC-based strain sensors have been developed and have demonstrated a wide strain range, high sensitivity, and good repeatability.

In terms of current flexible strain sensors in practical applications, there are still many areas for improvement. In the future, the production costs for reducing carbon-based fillers need to be further reduced, effective communication between the sensors and other electronic devices needs to be established, and the sensors need to be given the ability to accurately analyze and capture strain signals under complex environmental conditions. In summary, high performance carbon-based materials, optimized structural design, manufacturing methods, and working mechanisms pave the way for the preparation of carbon-based strain sensors with excellent sensing performance. When the current challenges are overcome, we expect CPC-based strain sensors to show greater potential for health monitoring, wearable electronics, human–computer interaction, and more.

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