

Application of graphene-silk protein composite material in wearable devices with its high sensitivity characteristics

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ABSTRACT

This paper reviews the research status of graphene-silk protein composites, focusing on their applications in the field of wearable device pressure sensing. Graphene has excellent electrical conductivity and mechanical properties, but poor biocompatibility and dispersibility. After compounding with silk protein, these problems can be solved, and the application value of the material is improved. A variety of preparation methods of composites were reviewed and their advantages and disadvantages were analyzed. The characterization shows that the composite has high strength, high fracture strain, good flexibility and excellent electrical and chemical sensing properties, and can respond quickly and maintain stability. It has development potential in the fields of health monitoring, motion tracking and environmental perception, and has good stability under extreme conditions, suitable for wearable devices. However, the material is prone to signal attenuation under repeated cyclic loading, and the cost of large-scale preparation is high, which limits its wide application. Future research should focus on developing low-cost manufacturing processes, optimizing durability and environmental adaptability, and expanding its application in medical health and smart devices.

KEYWORDS

graphene; silk protein; composite materials; wearable devices; performance characterization

1 Introduction

As the focus of development in the field of new materials, graphene is moving from laboratory to industrialization, and its market size is constantly expanding. In order to solve the problems of poor biocompatibility and difficulty in uniform dispersion, combining graphene with silk protein not only retains the excellent electrical conductivity and mechanical properties of graphene, but also improves the biocompatibility and tunable degradability of the material. This kind of composite material can achieve sensitive monitoring of object deformation, body movement, etc. Graphene-silk protein composite not only improves the application range of silk protein, but also promotes the application of green chemistry in electronic manufacturing, which has important social and economic value. However, wearable devices will face effects such as temperature and humidity changes, human sweat corrosion and mechanical friction during use, which may lead to a degradation of the performance of graphene composite materials. Therefore, how to improve the stability of the material in complex environments and ensure that it maintains high sensitivity performance during long-term use is a current challenge.

2 Literature review

2.1 Performance and technical characterization of graphene-silk protein composites

2.1.1 Mechanical properties

Graphene-Silk composites exhibit excellent mechanical properties such as high strength (339 MPa), high fracture strain ($611 \pm 85\%$) and good flexibility.

Ling et al. first successfully prepared a uniformly dispersed graphene/silk composite suspension through an optimized dissolution strategy. This uniform dispersion is the basis for achieving excellent mechanical properties. Then, graphene and silk fibers are mixed uniformly through two different solvent systems (HFIP and FA/Ca²⁺), forming a stable composite suspension.

In terms of mechanical properties, graphene/silk composites exhibit excellent ductility. For example, a graphene/SF/Ca²⁺ film containing 10% graphene exhibits strain (elongation of break) of up to 611% during the stretching process, and even if the graphene content increases to 40%, its strain can still reach 202%. This high ductility is mainly attributed to the random curling conformation of silk molecules and the water absorption capacity of Ca²⁺ ions. Ca²⁺ ions soften the material by absorbing moisture from the atmosphere, thereby significantly improving its toughness. In addition, graphene/SF composites exhibit significant synergistic effects in mechanical properties. When the graphene content is 25%, the composite exhibits optimal mechanical properties, with a strength of 242 MPa, a modulus of 4 GPa, and an elongation of break of 8%. These properties are superior to most traditional polymer composites. At the same time, the

toughness of graphene/SF/Ca²⁺ composite material is excellent.

Table 1 The toughness exhibited by composite materials with different graphene contents

Composite Material with 10% Graphene Content	Exhibits Toughness of 3.3 MJ/m ³
Composite Material with 20% Graphene Content	Exhibits Toughness of 13.9 MJ/m ³
Composite Material with 40% Graphene Content	Exhibits Toughness of 10.3 MJ/m ³

These data indicate that the composites maintain high toughness even at high graphene content ^[1].

Yang et al. used solution mixing method to prepare silk fibroin (SF)/graphene oxide (GO) composite material, and characterized the molecular chain conformation, mechanical properties and micromorphology of the composite material through scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and dynamic mechanical analysis (DMA)^[2].

Table 2 Characterization results of composite materials using multiple analytical methods

Analysis Method	Effect of GO Doping	Increase in GO Content
FT-IR Analysis	Promotes the transition of SF molecular chains from random coiling to β -sheet conformation	The content of β -sheet first increases and then decreases. When the mass fraction of GO is 1.0%, the content of β -sheet reaches its maximum value (33.1%)
XRD Analysis	Promotes the transition of SF from silk I type (mainly random coiling and α -helix) to silk II type (mainly β -sheet)	When the mass fraction of GO is 1.0%, the characteristic peak intensity of silk II type reaches its maximum
DMA Analysis	Significantly improves the tensile storage modulus (E') of SF composite material	When the mass fraction of GO is 1.0%, the E' of the composite material reaches its maximum value (about 5668 MPa), which is 157% higher than that of pure SF film. As the content of GO further increases, E' gradually decreases. The loss modulus (E'') of the composite material gradually decreases with the increase of GO doping, indicating that the doping of GO reduces the toughness of the composite material, but the overall mechanical properties are enhanced.
SEM Analysis AFM Analysis	The dispersion of GO in the SF matrix has an important impact on the mechanical properties of the composite material	When the content of GO is low, the GO sheets are uniformly dispersed in the SF matrix, which helps to improve the mechanical properties of the composite material; when the content of GO is too high, the GO sheets agglomerate, leading to a decrease in the mechanical properties of the composite material

Wang et al. pointed out that by feeding single-walled carbon nanotubes and graphene in silkworm cocoons, the mechanical properties of silk protein fibers can be enhanced. This reinforcement not only improves the fracture toughness and tensile strength of the fiber, but also improves its electrical conductivity. In addition, the introduction of graphene also changed the microstructure of the fiber, making it closer to the β -sheet conformation, thereby further improving the mechanical properties ^[3].

Liang et al. mentioned that by chemically reducing graphene oxide and combining with silk fibroin, a "brick-mud" layered composite film with excellent mechanical properties was prepared. This composite film not only demonstrates good mechanical properties and biocompatibility, but also shows that the addition of graphene significantly improves the overall performance of the composite material ^[4].

Yang, Ding et al. and Zhang also pointed out that the graphene/silicon conductive scaffolds prepared through electrospinning technology and the high-concentration graphene oxide/silicon fibre protein (RSF) fiber felt both showed significant improvement in mechanical properties. These studies further demonstrate the potential of graphene in enhanced silk protein composites ^[5-6].

Through stress tests, Xia et al. found that the addition of GO significantly improved the compression strength of the composite gel. Compared with the blank group, the compression strength of the GO variable group was increased by 40%, with the 0.2 wt% GO group showing the best compression performance and toughness. The addition of GO significantly enhances the mechanical properties of silk fibroprotein (SF) gel, which is mainly achieved through the following three mechanisms: First, more crosslinking structures are formed between GO and SF, and these crosslinking points not only increase the strength of the material, but also improve its toughness; secondly, the bridge structure of GO can effectively disperse stress and avoid stress concentration, thereby significantly improving the compressive resistance of the material; finally, the addition of GO also enhances the van der Waals force and hydrogen bonding between SF molecular chains, further improving the overall strength of the material^[7].

The high sensitivity of graphene-filament composites in strain sensors is mainly due to the piezoresistive effect of graphene. For example, it is mentioned in the literature that graphene/filament protein composites exhibit good resistance response under different strain conditions, and their sensitivity increases with the increase of external strain. Furthermore, the material was tested multiple cycles at maximum strain up to 200%, and the electrical response remained stable after 150 cycles, indicating its reliability in dynamic monitoring^[8].

In Wei et al.'s research, the mechanical properties of the composite film were significantly improved by using polydimethylsiloxane (PDMS) as the elastomeric matrix material. PDMS itself has excellent elasticity and flexibility. After being embedded in the composite film, the material can withstand more than 80% tensile deformation, while giving the composite film good stretching ability and reusability, effectively protecting the sensor from being easily destroyed during use and extending its service life. In addition, good compatibility between nanocellulose (CNF) and PDMS matrix enables the two to form a tight interface bond at the microscopic level. A reduced graphene oxide (rGO)-CNF film with a porous structure was formed on the PDMS substrate by solvent volatilization, and liquid PDMS was embedded in the pores of the film to cure, and finally formed a layered structure. This structure not only avoids the deformation inconsistency between the graphene nanosheet and the substrate due to insufficient adsorption force, but also further enhances the overall stability of the composite material. At the same time, by optimizing the preparation process, such as controlling the doping amount of rGO and CNF, the thickness of PDMS and the curing conditions, the mechanical properties of the composite can be adjusted so that it can better meet the practical application needs^[14].

2.1.2 High sensitivity and stability

Liu studied the sensitivity of the FSG strain sensor in detail through experimental and theoretical analysis. The contact area change mechanism is one of the key factors in high sensitivity. The sensor exhibits a linear response in the range of 0% to 60% strain with a sensitivity coefficient (GF) of 16.2; while at greater than 60% strain, the GF significantly increases to 150. This high sensitivity is derived from the overlap area changes and crack propagation effects of the graphene sheet layer. In the range of small strain (60%), the generation of cracks further reduces the conductive path and significantly improves the sensitivity. In addition, the authors also demonstrated that the FSG strain sensor has higher sensitivity over a wide strain range by comparing with other graphene-based strain sensors in the literature.

The FSG strain sensor exhibits excellent stability and repeatability. During the test of 5000 cycles stretching to 10% strain, the relative resistance changes of the sensor remained stable, showing an extremely low hysteresis effect. This stability is attributed to the low coefficient of friction between the graphene sheets and the mismatch of stiffness with the elastic tape, allowing the sensor to reversibly return to its initial state during stretching and release. In addition, cyclic testing at high tensile rates further verifies the stability of the sensor under dynamic conditions, indicating that it has reliable long-term performance in practical applications^[9].

Zhou et al. mentioned that graphene and Ecoflex-based composites were tested multiple cycles at a maximum strain of 200%, and their electrical response remained stable in all tests and showed good durability under repeated loading. This further demonstrates the cyclic stability of graphene-based composites in strain sensors^[8].

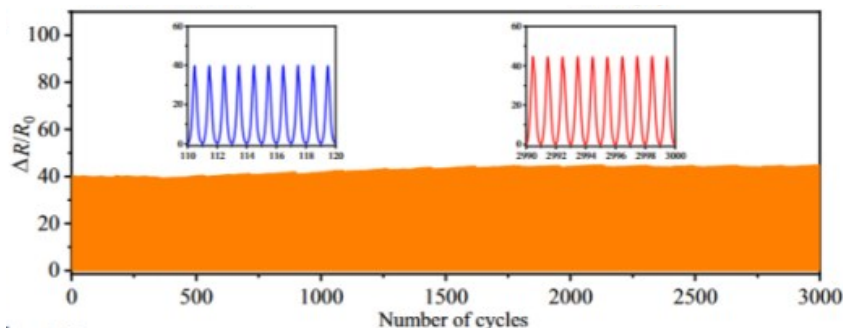


Figure 1 Cycle durability curve of strain sensor at 200% strain^[8]

To evaluate sustainability and long-term stability, Zhou et al. under the condition of maximum strain $\epsilon = 200\%$, performed 3,000 repetitions of the same device at a stretch-release scanning speed of 100 mm/min. Figure 1 shows that the electrical response is repeatable throughout the fatigue test, with only slight fluctuations, indicating good repeatability, stability and durability.

Liu et al., a graphene/silicon nanofiber membrane (SF/GR) prepared by electrospin, encapsulated in a pressure sensor made in PDMS, maintaining stable sensitivity under 2,000 cycle pressure loads. This shows that the composite material has good stability during long-term cycles^[11].

Wei et al. mentioned a TPU/graphene strain sensor, which exhibits strain sensitivity up to 400% and has good cycling stability. This new TPU/graphene strain sensor adopts a unique interleaved coil structure, showing 1100% tensileability, a sensitivity of 31.35 at 400% strain, and good cycling stability. Even if stretched to 400%, the sensor can still recover, which performance exceeds the limit of human movement^[10]. Although this study does not directly mention silk proteins, combined with the composite properties in the article, it can be inferred that graphene-silicon composites also have similar advantages in cyclic stability^[11].

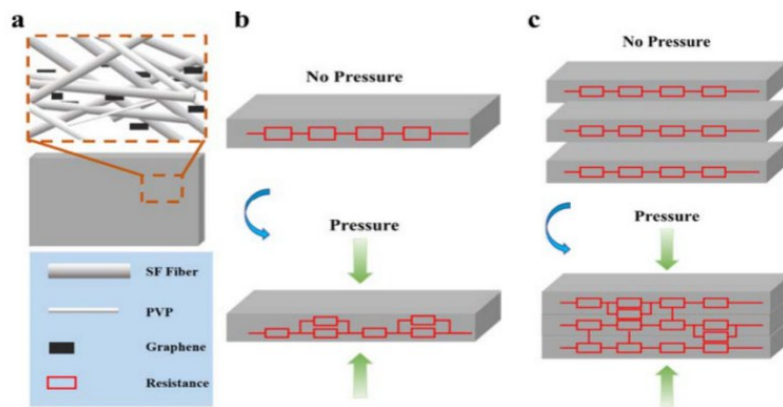


Figure 2 Working principle of SF/Gr pressure sensor [11]

2.1.3 Performance performance at extreme temperatures

Graphene-Silk composites are very sensitive to changes in humidity, temperature and chemical environments and can be used to monitor physiological parameters or environmental changes in the human body.

The performance of graphene-silicon composites under extreme temperature conditions is one of the key points of research. Graphene itself has extremely high thermal conductivity (about 5000 W/mK), which allows it to maintain stable sensing performance under high temperature environments^[12]. The thermal performance measurement of the silk fibroin membrane prepared by electrospinning was performed by a thermal conductivity tester. The results showed that the thermal conductivity of the silk fibroin membrane was low (0.0959 W/m·K), indicating that it had good thermal insulation properties. This low thermal conductivity characteristic allows it to effectively isolate external temperature interference and is suitable as a flexible substrate material for temperature sensors^[13].

Wei et al. optimized the microstructure of composite materials and used elastic groups. In this study, the porous structural properties of nanocellulose (CNF) played a key role in the moisture resistance of composite materials. CNF intersects and overlaps with each other in the composite material to form a porous structure. This structure not only gives the composite material good breathability and water absorption, so that it can quickly absorb and release moisture, reduce the accumulation of moisture inside the material, but also significantly improve the material's moisture resistance. In the water absorption experiment, the rGO-CNf film has a rich pore structure and can quickly absorb moisture; while the pure rGO film is relatively dense and difficult to immerse in moisture. This further demonstrates the important role of porous structures in improving wet resistance. In addition, by optimizing the dispersion of rGO and CNF, a stable conductive network structure is formed inside the composite material. This structure not only improves the electrical properties of the material, but also enhances its moisture resistance, because a stable conductive network can reduce the interference of moisture on electrical properties, thereby improving the stability of the material in humid environments^[14].

In the range of 25°C to 80°C, strain sensors based on MWCNTs and H-70.30 materials are able to maintain stable performance, with scaling factors varying from 3 to 2.3, indicating their stability under temperature changes. Figure 2 shows the relative changes in the resistance of thin film sensors manufactured under strain. Graphene showed higher sensitivity to strain with a relative change of 12.4%, while MWCNT and mixed samples had a relative change of 1.1% and 3.7%, respectively. The resistance exhibited by the mixed samples was estimated to have a strain coefficient of graphene 42.7, MWCNT 4.63, and a hybrid nanocomposite H-70:30 of 16.21. Although mixed samples exhibit less strain coefficient

than graphene, their key advantage is their ability to operate as a strain sensor in the temperature range of 25°C to 80°C without significant influence from temperature changes. In addition, the mixed samples experienced cyclic strain of 100 cycles; the response was shown in Figure 3, and the sensor response changed from 3% to 2.3% in the initial cycle and then stabilized. Despite the slight fluctuations, the sensor is able to respond with good linear induction over 100 cycles.

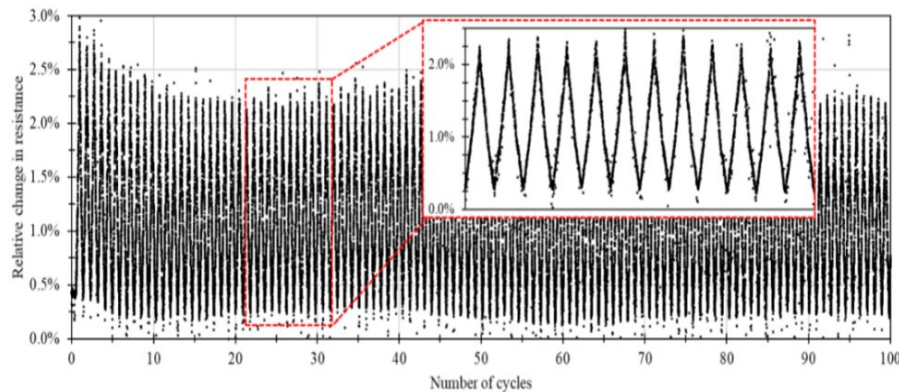


Figure 3 Comparison of strain coefficients of thin film strain sensors and the response of mixed samples under cyclic strain loading test ^[15]

2.2 Application Status and Development Field of Graphene-silk Protein Composites

Graphene-silk protein composites combine the high strength and high conductivity of graphene with the biocompatibility and biodegradability of silk protein, and have certain application achievements at this stage.

By utilizing the property that silk protein can selectively aggregate on reduced graphene oxide sheets, a mixed solution of reduced graphene oxide sheets and silk protein with silk protein microfibers enriched on reduced graphene oxide sheets and good dispersibility was prepared, and a series of reduced graphene oxide/silk protein-based porous composites with different proportions were obtained by freezing and low temperature ethanol treatment. Reduced graphene oxide/silk protein-based porous composites have good strength and elasticity under completely wet state. Because of their high sensitivity, wide working range, adjustable structure and good plasticity, they are expected to obtain better applications in flexible pressure sensing ^[16].

Graphene-silk protein composites have also demonstrated their potential in contactless wearable devices. For example, a smart sensor using silk fibroin reduced graphene oxide (SF-rGO) as the sensing active layer enables highly sensitive sensing of humidity and temperature over a wide linear range and accurate correction of thermal readings by a Joule heater ^[1]. This sensor can be used for non-contact sensing applications such as respiratory monitoring and mental state prediction. The researchers have also developed silk protein-graphene composite chemiresistive smart sensors for contactless wearable devices, demonstrating their potential for health monitoring and making them ideal for dynamic deformation monitoring due to their high sensitivity and cyclic stability ^[17].

By combining graphene oxide with silk protein, the mechanical properties of composites can be significantly improved, such as compressive strength increased by 41% and Young's modulus increased by 75% ^[18].

The composite material can be used to manufacture intelligent pressure-sensitive devices, supercapacitors, flexible sensors, pressure sensors and multifunctional wearable devices, and its high sensitivity and stability make it important for health monitoring, environmental monitoring and human-computer interaction. ^[19] The biodegradability of silk protein combined with the high strength of graphene makes the composite a green and sustainable new material ^[18].

3 Research technique

Through academic databases such as China Knowledge Net, Wanfang Data Resources and Scihub, academic literatures at home and abroad on graphene, silk protein, composite materials of graphene and silk protein and their application in wearable pressure sensing field are widely collected to understand the recent research progress and application of graphene-silk protein composite materials. Literature management tools are used to classify the collected literature into different aspects according to the research content, such as synthesis and characterization, performance technology characterization, high sensitivity pressure sensing, application field, etc.

The preparation methods of graphene-silk protein composites, including chemical vapor deposition, solution mixing, and in-situ polymerization, are reviewed in the literature. The strengthening mechanism of these composites for wearable devices is outlined.

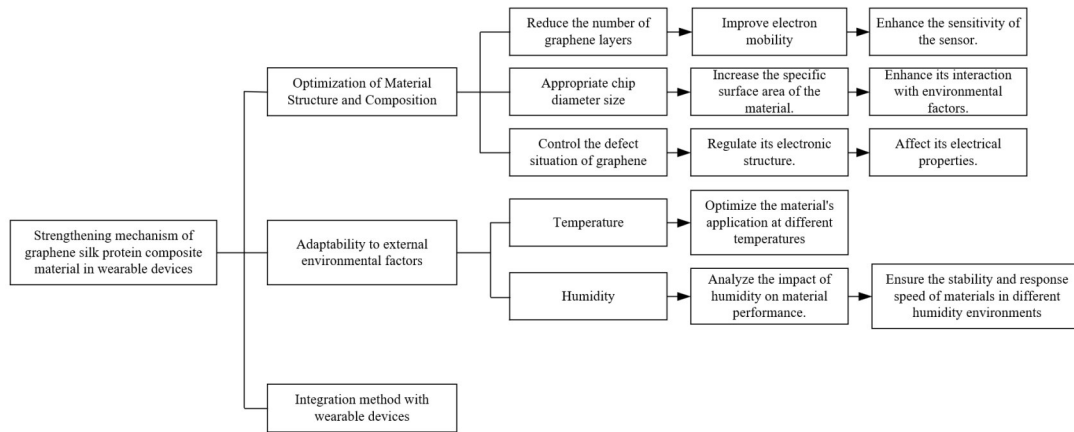


Figure 4 Strengthening mechanism of graphene silk protein composite material in wearable devices

Graphene-silk protein composites show broad application prospects in wearable devices. It combines the electronic advantages of graphene with the mechanical properties of silk protein, has ultra-high stretchability and strength, and is suitable for manufacturing wearable sensors, smart textiles and smart skin. The material not only withstands frequent stretching and bending, but also maintains stability and sensitivity under extreme conditions. In addition, it can be used to manufacture electromyography (EMG), pressure sensors and chemical resistance sensors, which are widely used in health monitoring, motion tracking and human-computer interaction.

4 Discussion

4.1 pattern analysis

4.1.1 Preparation and Properties Optimization of Composites

The existing research generally focuses on the preparation methods of graphene-silk protein composites (such as solution mixing method, in-situ polymerization method) and their performance optimization strategies (such as reducing the number of graphene layers and controlling the defect distribution). Researchers tend to improve the mechanical properties and electrical sensitivity of materials through structural design (such as "brick-clay" layered structure) and functional treatment (such as introducing elastic matrix PDMS). Microstructural adjustment (porous design, dispersion optimization) is a typical mode to improve macro-performance.

4.1.2 multidimensional performance characterization

Electrical, mechanical, and chemical sensing properties (e.g., electrical resistance change, tensile test, gas response) have been extensively used in the literature to systematically evaluate composite properties. Cyclic stability testing (e.g. 5000 tensile cycles) has become the standard method for verifying material durability. Most often, a combination of experimental and theoretical analysis (e.g., contact area variation mechanisms) explains the source of high sensitivity.

4.2 trend analysis

4.2.1 From Single Material to Functional Composite Material

The current research trend is clearly turning to composite materials to integrate the electronic advantages of graphene with the biocompatibility of silk proteins. For example, the versatility can be extended by introducing an elastomeric matrix (PDMS) or nanocellulose (CNF) to enhance moisture resistance, or by combining other nanomaterials such as MXene. According to the existing research, it is speculated that the development of smart composites with self-healing and environmental response (such as temperature and humidity adaptation) is a hot topic in the future.

4.2.2 From laboratory research to practical application drive

The research goal gradually shifts from basic performance exploration to practical application requirements, and people pay more and more attention to health problems in today's society. Therefore, the development of health monitoring equipment based on this composite material can be regarded as the development goal. In addition, the

design of flexible sensors that can detect harmful gases or temperature/humidity changes is also one of the current hot spots. The development of environmentally sensitive equipment can be used as detection equipment for people's daily travel and can also be combined with medical treatment. Some patients may be sensitive to the air environment. This kind of equipment can help patients monitor from time to time and make timely adjustments in the face of adverse factors.

4.2.3 Green and sustainability orientation

Combined with the biodegradability of silk protein, it promotes the development of green electronic materials to reduce the impact of traditional electronic devices on the environment.

5 Conclusion

Graphene and silk protein composites can be prepared by various methods, including chemical vapor deposition, solution mixing and in-situ polymerization. Each of these methods has its advantages and disadvantages as shown in the table below.

Table 3 Advantages and disadvantages of three preparation methods for composite materials

Preparation method	Advantage	Disadvantage
Chemical Vapor Deposition	Ability to prepare high-quality graphene	it has high costs and complex processes.
Solution mixing method	Easy to operate	it may lead to the aggregation of graphene, thereby affecting the performance of the material.
In-situ polymerization	It is possible to directly form a silk protein layer on the surface of graphene, demonstrating good dispersibility and stability.	It is necessary to precisely control the reaction conditions, such as temperature and pH levels. Additionally, the reaction process may generate by-products, which can affect the purity and performance of the final product.

By reducing the number of graphene layers, optimizing the sheet diameter and controlling the defects of graphene, the electronic structure of graphene can be adjusted, which in turn affects its electrical properties, thus enhancing the sensitivity of the sensor. Based on these optimization strategies, graphene-silk protein composites exhibit extremely high sensitivity in strain sensors, such as strain detection limits as low as 0.025%, and good cycle stability (over 1000 cycles), making them very suitable for human motion monitoring and health detection. In addition, the composite material is very sensitive to humidity, temperature and chemical environmental changes (high sensitivity and stability), and can be used to monitor human physiological parameters or environmental changes, further expanding its application potential in wearable devices, motion tracking and environmental monitoring.

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