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Article

# Flexible and Stretchable MXene/Waterborne Polyurethane Composite-Coated Fiber Strain Sensor for Wearable Motion and Healthcare Monitoring

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**Abstract:** Fiber-based flexible sensors have promising applications potential in human motion and healthcare monitoring, owing to their merits of lightweight, flexibility, and easy processing. Now, high-performance elastic fiber-based strain sensors with high sensitivity, large working range and excellent durability are in great demand. Herein, we have easily and quickly prepared a highly-sensitive, durable fiber-based strain sensor by dip-coating of highly-stretchable polyurethane (PU) elastic fiber in MXene/ waterborne polyurethane (WPU) dispersion solution. Benefiting from the electrostatic repulsion force between the negatively charged WPU and MXene sheets in the mixed solution, very homogeneous and stable MXene/WPU dispersion was successfully obtained and the interconnected conducting networks were correspondingly formed in coated MXene/WPU shell layer, which makes as-prepared strain sensor exhibit a gauge factor of over 960, a large sensing range of over 90% and detection limit as low as 0.5% strain. As elastic fiber and mixed solution have the same polymer constitute, tight bonding of MXene/WPU conductive composite on PU fibers was achieved, enabling the as-prepared strain sensor endure over 2500 stretching-releasing cycles and thus show good durability. Full-scale human motion detection was also performed by the strain sensor, and a body posture monitoring, analysis, and correction prototype system was developed via embedding the fiber-based strain sensors into sweater, strongly indicating the great application prospect in exercise, sports and healthcare.

**Keywords:** MXene; polyurethane; fiber; dip coating; strain sensor; motion detection; body posture; healthcare

## 1. Introduction

Flexible strain sensors have received great attention due to their high efficiency in converting mechanical deformations of the human body into electrical signals, enabling their broad applications in human motion detection, sports, real-time healthcare monitoring, etc [1–5]. Among all types of stretchable substrates needed for flexible sensors, fibers are one of competitive choice as their virtues of lightweight, flexibility, and embeddability, which allows for the seamless connection with fabrics and cloth [6–10]. Now, simple, mass, and reproducible fabrication of fiber-based flexible strain sensors with excellent performance is well required. For such intention, several methods such as wet-spinning [11–13] and dip-coating [14–16] have been reported. For example, Huang et al. developed a coaxial fiber-based strain sensor with high sensitivity and wide detection range by a continuous, facile, and scalable wet-spinning approach [17]. However, wet-spinning process generally includes the formation of fine stream by pressing as well as the production of fibers by coagulation in the solution, which is often completed by the specific equipment or apparatus. Dip coating is another

widely-adopted facile approach, which normally deposits conductive layer on fiber surface, thus holding the advantage of high efficiency and low equipment requirements over wet-spinning. For instance, in our previous work, fiber-based strain sensor were fabricated by simply hydrothermal reducing the dip-coated graphene oxide on elastic fibers of yarns, human motion monitoring, sports monitoring, healthcare monitoring and gesture recognition have been successfully demonstrated by them.[18–21] Nevertheless, we find that dip-coated solid conductive materials have relatively poor adhesion to the fiber and mechanical properties mismatch exists between them, which makes solid coating layer difficult to endure daily rubbing or numerous stretching-releasing cycles of the fiber sensor in actual usage scenario. Considering the robustness, adhesiveness, controllable electrical properties and mechanical properties of dip coating layer, conductive polymer composites (CPCs) which consist of conductive filler and polymer matrix have become an ideal substitute [22–26].

Recently, a new large family of two-dimensional (2D) transition metal carbides or nitrides, called MXene, has become a very-hot conductive filler for CPCs and gradually shown the superiority to the counterparts already-used by strain sensors due to its metallic conductivity and high specific surface area [27–31]. Moreover, abundant polar functional groups generated during the liquid-phase etching process endow MXene with excellent hydrophilicity, enabling and facilitating the solution-based employment approaches of them [32]. In addition, with the rise of environmental awareness, the use of non-pollution, low-toxicity polymer has become another trend for CPC [33–35]. Waterborne polyurethane (WPU), a new type of PU system that uses water instead of organic solvents as dispersion medium, has abundant hydrophilic functional groups, so it can form a strong bond to various substrates through hydrogen bonding, showing excellent film-forming properties, processability and thus attracting much attention [36–38]. However, MXene nanoflakes or nanosheets dispersion are susceptible of stacking in WPU solution, which makes it difficult to construct uniform and stable CPCs dispersion. Fortunately, such dilemma can be effectively removed or suppressed by introducing electrostatic repulsion mechanism into MXene/WPU composites [39–41], i.e., negatively-charged WPU was chosen to mix with negatively-charged MXene. At the same time, if PU elastic fiber was accepted for dip coating, fiber and CPC dispersion will have the same PU polymer constituent and the mechanical mismatch of Young's modulus between fiber and CPC will be significantly reduced, thus very strong bonding of CPC and PU fiber can be expected.

Herein, homogeneous MXene/WPU composite dispersion was prepared by blending negatively-charged WPU solution and MXene nanosheets solution, which was then dip-coated on the PU elastic fiber matrix to prepare elastic fiber-based strain sensor. As-prepared fiber strain sensor exhibited attractive sensing performance, including high sensitivity with a gauge factor of over 960, a broad sensing range of over 90%, and a low detection limit of 0.5%, thus full-scale human motion detection from vigorous joint movement to subtle expression change and wrist pulse was realized. Moreover, benefiting from the tight bonding between MXene/WPU composite coating layer and PU fiber, the fiber strain sensors exhibited good washing and peeling resistant capability as well as good durability of over 2500 stretching-releasing cycles, which is robust enough to endure daily human activities. Finally, a smart data glove for finger bending angle and hand gesture monitoring and a prototype body posture monitoring and correction system were developed via embedding the fiber-based strain sensors into common fabrics, demonstrating the great potential of as-fabricated wearable, comfortable, non-intrusive sensors in exercise, sports, gesture recognition and healthcare applications.

## 2. Materials and Methods

### 2.1. Materials

The  $\text{Ti}_3\text{AlC}_2$  MAX phase (400 mesh) was purchased from Ji Lin 11 Technology Co., Ltd, China. Lithium fluoride (LiF, 99.9% purity) and hydrochloric acid (HCl, 37% concentration) were purchased from Aladdin Reagent Co., Ltd, China. WPU (35 wt%) with good adhesion (300 mpa·S in viscosity) was purchased from Shenzhen Jitian Chemical Co., Ltd, China. PU elastic fibers (~500  $\mu\text{m}$  in diameter) were purchased from Jiang Su Geruite Textile Co., Ltd, China.

## 2.2. Synthesis of MXene nanosheets suspension

40 ml of 9 M HCl acid and 2 g LiF powder were slowly added into a Teflon container. Magnetic stirring was employed to obtain a sufficient dissolution and mixing. Then 3 g of  $Ti_3AlC_2$  MAX precursor powder was slowly added into the above mixed solution for etching and the whole etching procedure was kept 24 hrs at 35 °C. After full reaction and etching, the obtained suspension was centrifugally separated (4500 rpm, 4 min) and washed with deionized water for several times until the pH value reached about 6. After that, the obtained black sediment was diluted with an appropriate amount of deionized water and delaminated under sonication. Then the mixture was further centrifugally separated (3000 rpm, 30 min), and the dark green middle solution was collected as the MXene nanosheets suspension. The typical fabrication steps of  $Ti_3AlC_x$  MXene from  $Ti_3AlC_2$  MAX precursor were shown by SEM images and XRD pattern in Figure S1.

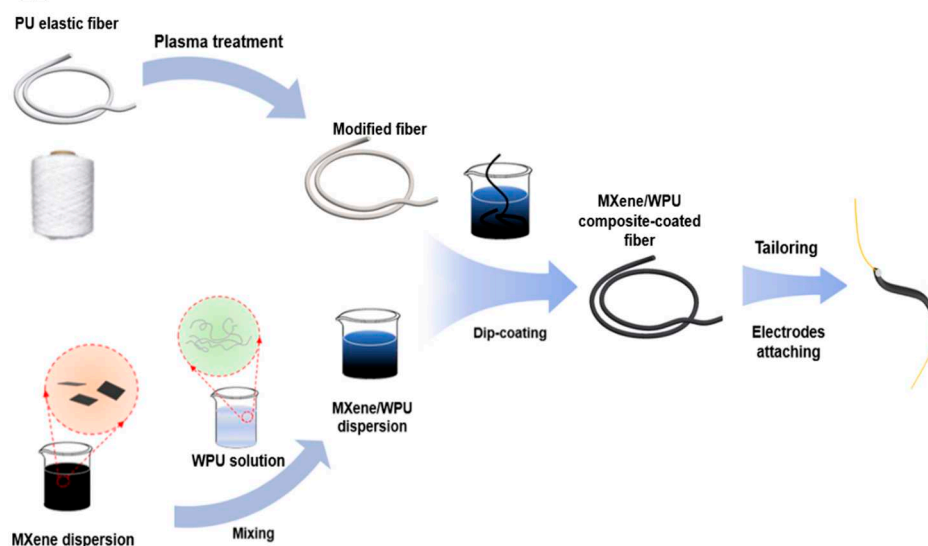
## 2.3. Preparation of MXene/WPU composite-coated fiber sensor

MXene/WPU dispersion solution with various MXene/WPU mass ratio (1:1, 1:3, 1:5 and 1:7) was obtained by adding negatively charged WPU (PU, 35 wt%) solution into specific amount of  $Ti_3C_2T_x$  MXene suspension. To get fully mixed, MXene/WPU solution was then magnetic stirred for 1 hr at room-temperature.

PU elastic fibers were washed thoroughly by ethanol and DI water for 5 mins, respectively and then transferred into an oven to get dry at 50 °C for 1h. Next, the elastic fibers were treated by glow discharge air plasma for 10 minutes to increase the surface roughness and generate polar groups [42]. Subsequently, the elastic fibers were dipped into MXene/WPU dispersion solution for a 15 min ultrasonic treatment, and transferred into the oven to get dry at 50 °C for 1 h again. After drying, CPC layer was successfully coated on the surface of elastic fibers. A series of MXene/WPU composite-coated elastic fibers were fabricated and cut into short section with equal length of 4 cm. With the assistance of conductive silver glue, both ends of the tailored fibers were connected with 5 cm copper wire electrodes, finally forming the fiber-based strain sensor.

## 2.4. Characterization

Zeta potentials of  $Ti_3C_2T_x$  suspensions and WPU were determined by a nanoparticle size and zeta potential analyzer (Zetasizer Nano ZS90, Malvern, UK). The morphologies of MXene/WPU composite coating layer were observed using field emission scanning electron microscope (Merlin, Carl Zeiss, Germany). The strain sensing performance of the MXene/WPU composite-coated fiber strain sensor were measured by a simple coupling system that consists of a universal testing machine (AG-XplusHS 1kN, Shimadzu, Japan), a source meter (B2902, Keithley, USA), and a computer. The cyclic test of fiber sensor was conducted by applying circulative strains through an algorithm-controlled stepper motor.

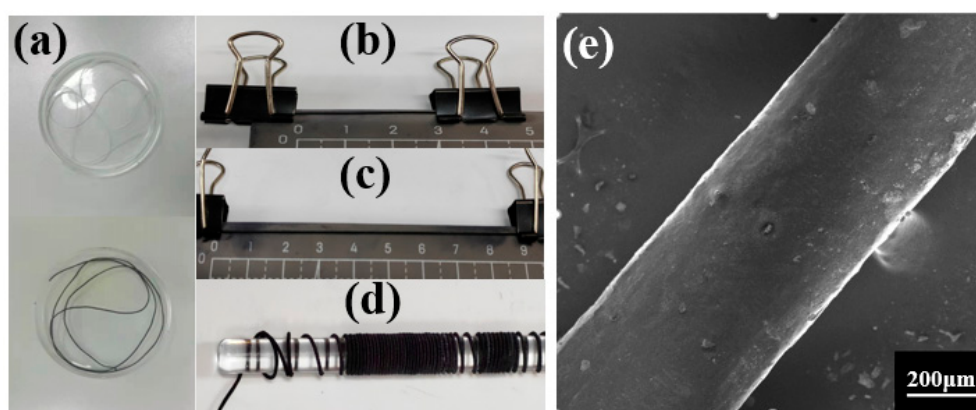


**Figure 1.** Schematics of the Fabrication flows of MXene/WPU composite-coated fiber sensor;

### 3. Results and Discussion

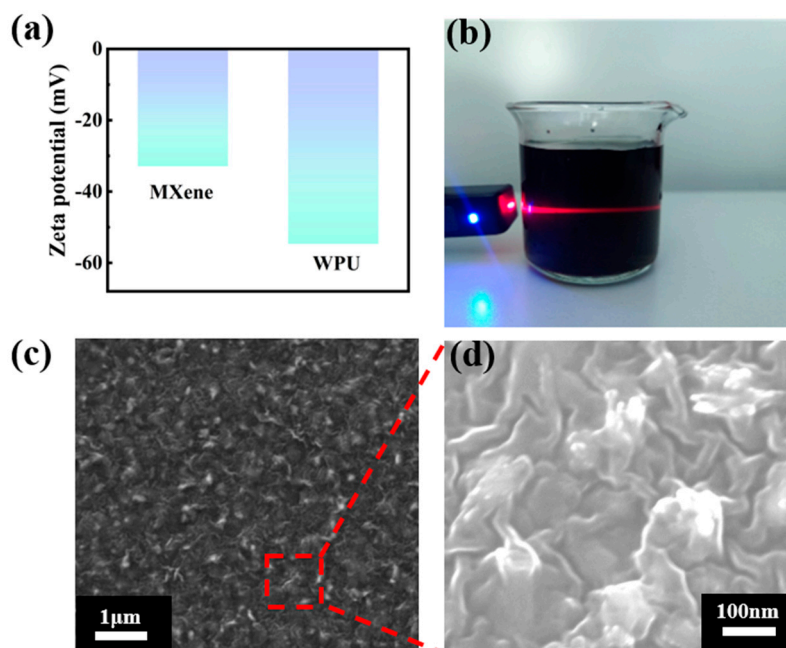
#### 3.1. Morphologies of MXene/WPU composite-coated fiber

After one time dip-coating process, MXene/WPU composite conductive layers were successfully coated on the surface of PU elastic fibers, accompanied by an obvious change of fiber color from white to black, as shown in Figure 2(a). As presented in Figures 2(b-d), the coated fibers endured a large strain of 300% and were also tightly wound on a thick perspex bar, showing good stretchability and favorable flexibility, respectively. Without any conductive treatment to the sample such as gold sputtering or spraying carbon powder, magnified SEM image of the coated fiber is obtained and shown in Figure 2(d), which indicates MXene/WPU CPC layer has excellent conductivity. Moreover, the coated fiber held a uniform cylindrical structure and smooth surface, which is beneficial to the future weaving or application of the coated fibers.



**Figure 2.** (a) Photographic comparison of untreated fiber and MXene/WPU composite-coated fiber; Photographs of MXene/WPU composite-coated fiber (b) without any strain, (c) under 300% strain and (d) wound on a perspex bar. (e) SEM image of MXene/WPU composite-coated fiber.

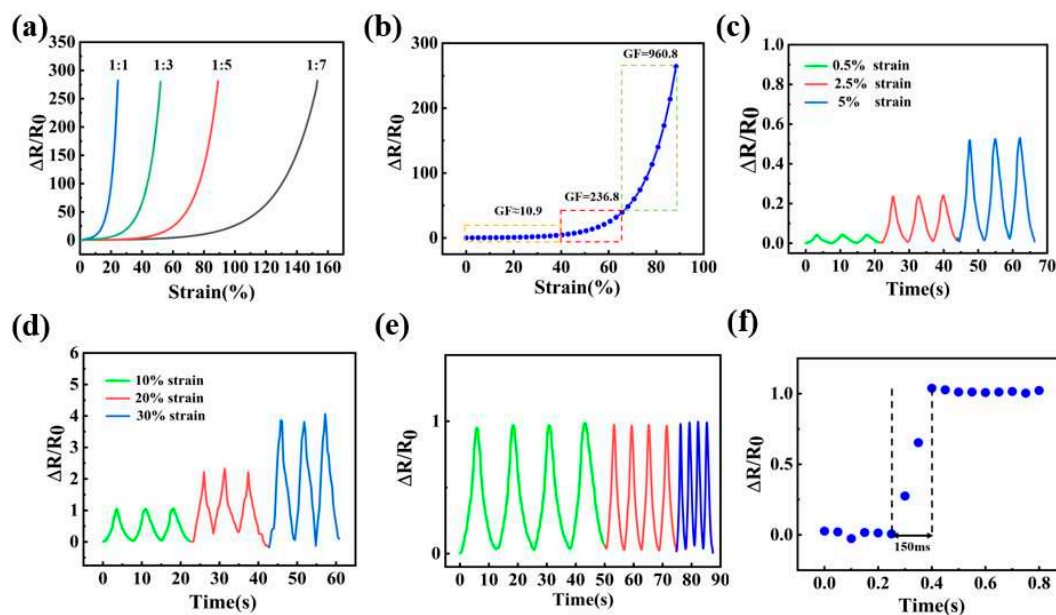
As indicated by the measured zeta potentials (Figure 3a), both MXene nanosheets and WPU molecule have adequate negative charges, which guarantees the formation of homogeneous MXene/WPU dispersion under the electrostatic repulsion between them. As shown in Figure 3b, very clear Tyndall effect was observed by naked eyes, providing the powerful evidence of homogeneous and stable dispersion of MXene nanosheets in WPU solution [43]. Furthermore, as shown in Figures 3(c-d), magnified SEM images of MXene/WPU composites show the uniform distribution of MXene nanosheets in the WPU polymer, which validates the homogeneous dispersion again.



**Figure 3.** (a) Zeta potentials of  $\text{Ti}_3\text{C}_2\text{T}_x$  and WPU solutions. (b) Tyndall effect observed from MXene/WPU dispersion. (c-d) SEM images of MXene/WPU composite layer coated on PU fiber.

### 3.2. Strain sensing performance of MXene/WPU composite-coated fiber

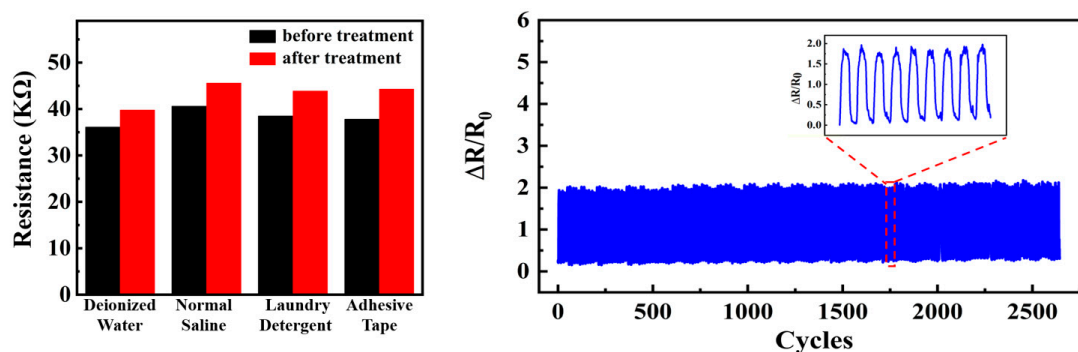
Firstly, resistance variation of MXene/WPU composite-coated fibers with different MXene content were systematically investigated under the continuously-increased strain is shown in Figure 4a. All the fiber samples displayed a resistance ramping trend along with the strain increment, which was ascribed to the destruction of the conductive network upon external tension. In addition, higher MXene content in the composite will induce a higher gauge factor (GF) of the fiber while the significant reduction of the stretchable range or the affordable strain. According to the overall strain sensing performance, MXene/WPU composite-coated fiber with MXene content of 16.7% (i.e., MXene/WPU mass ratio =1:5) was selected as the sole fiber sensor sample for the following tests. Figure 4b shows the resistance variation curve of the sensor sample under strain ranging from 0 to 90%, which can be further divided into three sections: 0-40% strain ( $\text{GF} \approx 10.9$ ), 40-65% strain ( $\text{GF} \approx 236.8$ ) and 65-90% strain ( $\text{GF} \approx 960.8$ ), the quickly-enhanced sensitivities was ascribed that much more conductive networks in the coated MXene/WPU composite were destroyed when great strain was exerted. Cyclic strain sensing behaviors of the sensor under tiny strain (0.5-5 % strain) and large strain (10-30 % strain) are comprehensively investigated and shown in Figures 4c and 4d, from which it can be clearly seen that relative sensor resistance ramps up with increasing strain and the resistance of the sensor almost completely returns to the initial value once releasing the strain, exhibiting outstanding reproducibility and stability. Furthermore, the electrical response of the composite-coated fibers to fixed strain of 10% under the frequency range of 0.08-0.32 Hz was investigated and shown in Figure 4e, from which the resistance variation of the sensor was negligibly affected by applied frequencies, thus indicating an excellent dynamic reliability. As shown in Figure 4f, the sensor shows a rapid response time of 150 ms when exerted quasi-transient step strain of 10%, making it feasible to be used in the fields such as human motion monitoring which generally requires timely feedback.



**Figure 4.** (a) Resistance variant curves of MXene/WPU composite-coated fibers with four MXene/WPU mass ratio under tensile strain and (b) Magnified curve for MXene/WPU mass ratio of 1:5. Cyclic sensing behaviors of the sensor under (c) tiny strain, (d) large strain, and (e) fixed 10% strain with variable frequencies. (f) Response time of the sensor to quasi-transient step strain (10% strain).

### 3.3. Robustness and durability of MXene/WPU composite-coated fiber

Three trial groups of same MXene/PU composite-coated elastic fiber sensor samples were immersed in DI water, saline solution, and laundry detergent solution, respectively, and all ultrasonicated for 1 h, simulating the exposure of the sensor sample to rain-washing, sweat soaking and washing in daily life. In addition, a piece of transparent adhesive tape was also tightly attached to the CPC layer and then suddenly peeled off, artificially creating the mechanical wear of sensor sample from rubbing. As shown in Figure 5 (a), the average resistance value of sensor sample only showed a slight change of <10% against above harsh treatments, showing adequate robustness of the sensor samples. Furthermore, the sensor sample was further evaluated by a cyclic stretching-releasing test (> 2500 cycles) under 20% fixed strain, as shown in Figure 5 (b), stable sensing response was clearly recorded, further showing good durability of the sensor samples.

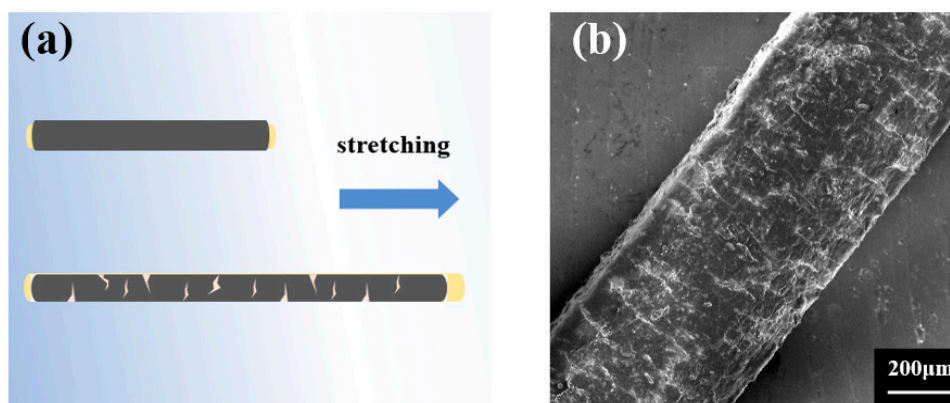


**Figure 5.** Resistance change of the sensor under (a) various treatments and (b) >2500 repeated stretching-releasing cycles.

### 3.4. Strain Sensing Mechanism Analysis

Sensing mechanism of the fiber strain sensor was revealed by investigating the structural change of MXene/WPU CPC layer during stretching-releasing cycle. As illustrated in Figure 6(a), the CPC shell layer was extended under sensor stretching, and obvious microcracks that lengthen the

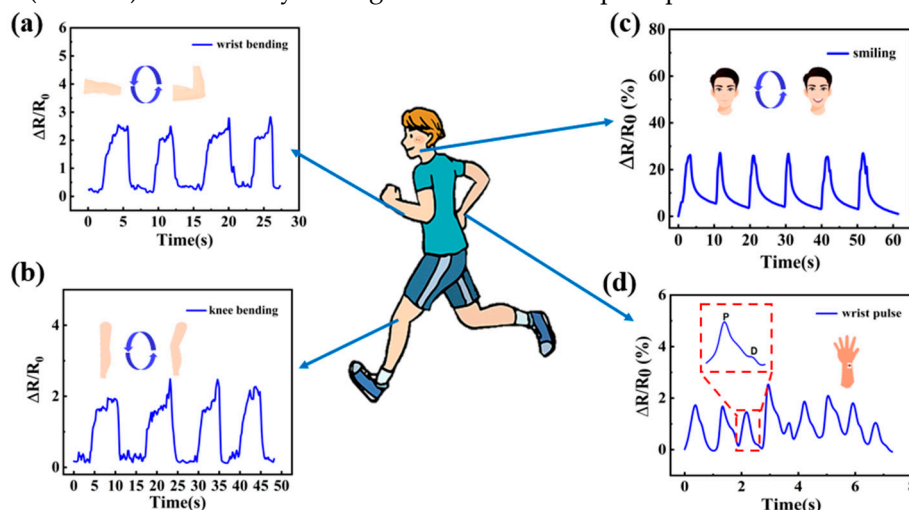
transmission pathway of the electrons and thus increase the resistance of the sensor will be generated in CPC layer, which were further verified by SEM image of the stretched fiber sensor in Figure 6(b). Upon releasing the sensor, the microcracks will close again, which makes the previous prolonged conductive pathways recover to the original state again and thus induces the resistance decrease [44,45]. Obviously, it is very reasonable to believe that the obtained strain sensing performance is ascribed to those microcracks.



**Figure 6.** (a) Schematic illustrations showing the sensing mechanism of the MXene/WPU composite-coated fiber (b) SEM image of the MXene/WPU composite-coated fiber under stretching.

### 3.5. Full-scale human motion detection

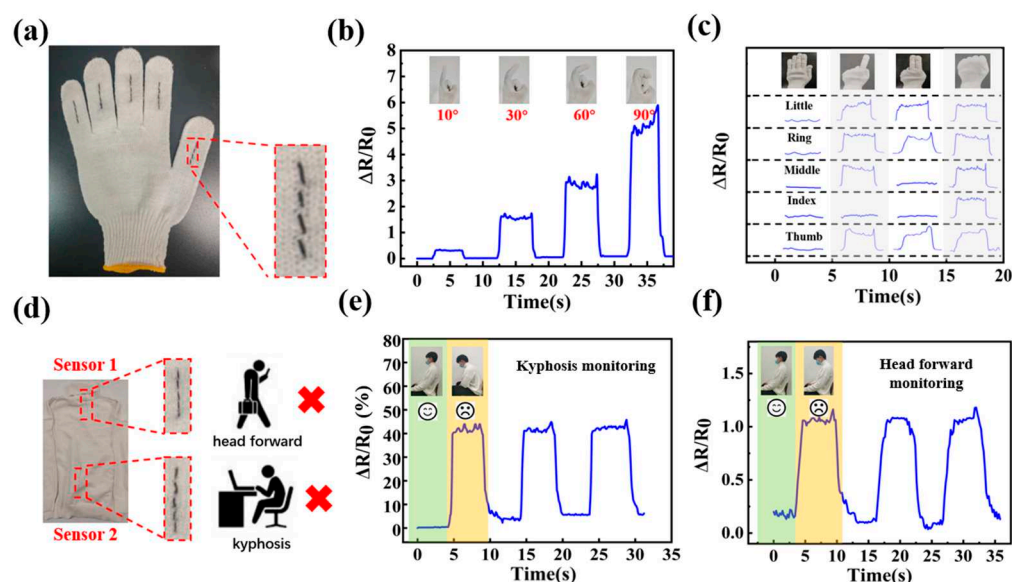
Favorable sensing performance obtained by the fiber strain sensor including very high sensitivity, wide working range, excellent robustness and long-term durability strongly enables them as the attractive candidates for wearable electronic applications. For the applicable validation, the sensor samples were directly mounted on the corresponding body parts of one young guy to continuously record the electrical signal waveform when he exerts full-scale human motions including intense human activities like joint movements and subtle human activities like expression and wrist pulse. As shown in Figure 7(a) and 7(b), a reproducible signal waveform was clearly recorded when the volunteer performed the bending and releasing movement of his elbow joint, and knee movement waveform when walking or running was also repeatedly exhibited, both demonstrating the ability of the sensor samples to detect the high intensity motions. As shown in Figure 7(c) and 7(d), thanks to the high sensitivity, the sensor mounted on the face successfully tracked the subtle facial expression changes from silence to smiling, the resistance of the strain sensor also varies regularly with the wrist pulse wave, additionally the percussion wave (P-wave) and the diastolic wave (D-wave) can be easily distinguished in one complete pulse wave.



**Figure 7.** Resistance variation curves of the fiber strain sensor when measuring (a) wrist bending, (b) knee bending, (c) smiling, and (d) wrist pulse.

### 3.6. Applications for Wearable Gesture and Healthcare Monitoring.

Benefiting from the good embeddability and processability brought by the fiber as typical 1D structure, elastic fiber-based strain sensor can be woven into various fabrics or cloth so as to achieve more advanced wearable sensing besides monitoring physiological condition and motion activities. As shown in Figure 8a, a flexible data glove was fabricated through sewing five discrete fiber strain sensors onto the articulation positions of one cotton string glove, every sensor can accurately monitor the bending angle of the corresponding finger joint. Taking the index finger as an example, as shown in Figure 8(b), different angular positions ( $10^\circ$ ,  $30^\circ$ ,  $60^\circ$  and  $90^\circ$ ) of the bending finger can be distinguished by the sewed fiber strain sensor. Furthermore, as illustrated in Figure 8(c), simple gestures made by the data glove can be readily recognized via comprehensively evaluating the intensity and patterns of the detected five-channel signals. Healthcare monitoring application of the fiber strain sensors was also investigated. As well-known already, poor sitting postures such as head forward and kyphosis will lead to many musculoskeletal disorders, therefore research on posture monitoring and correcting is of great importance and has been a hot focus. In this study, using the fiber strain sensors as the core sensing components, a simple prototype system has been successfully developed. In details, as shown in Figure 8d, smart clothing was prepared by sewing fiber strain sensors onto different parts (marked red) of the back region of a turtleneck tight sweater, in which sensor 1 and sensor 2 are used to monitor neck movement and back bending, respectively. The sensitive fiber sensors can detect the local deformations of the sweater caused by a sitting posture change, and the collected signals are then transmitted to an Arduino microcontroller for further processing and analysis in order to evaluate the postures, finally audible and light feedback will be given for personal prevention. As shown in Figure 7(e-f) and corresponding video (Supplementary Materials, video S1 and S2), poor body postures which cause an increase in sensor resistance including excessive head forwards and kyphosis (yellow part) were successfully detected by the system, and then a buzzer alarm was activated to alert the wearer.



**Figure 8.** (a) A smart data glove woven with five fiber strain sensors onto the joints of the five fingers. Electrical signal output of the fiber strain sensors on data glove were used for monitoring different (b) bending angles of each finger and (c) gestures performed by five fingers. (d) A prototype wearable system based on the smart clothing woven with two fiber strain sensors was developed. Electrical signal output of the fiber strain sensors were utilized for monitoring (e) kyphosis and (f) head forward.

#### 4. Conclusions

In this study, MXene/WPU composite-coated fiber strain sensors with favorable sensing performance were successfully fabricated by a facile and quick method, i.e., simply dip coating elastic PU fibers into homogeneous MXene/WPU dispersion. Electrostatic repulsion between negatively-charged MXene and WPU plays a critical role in the uniform dispersion of MXene nanosheets in WPU solution. Moreover, due to the abundant surface functional groups in MXene and WPU, together with the identical PU polymer constituent, very firm bonding was achieved between MXene/WPU conductive polymer composite and stretchable PU matrix, meanwhile very small Young's modulus mismatch existed between them, all of which endow the fiber strain sensors with super resistant capability to washing, peeling and cyclic stretching-releasing. Moreover, thanks to the high sensitivity and large strain working range, we investigated the applicability of such fiber strain sensor in human motion monitoring from vigorous joint movements, expression change to subtle wrist pulse. Finally, a smart data glove for finger bending angle and hand gesture monitoring and a prototype system for human body posture monitoring and correction were achieved by embedding the fiber strain sensors into ordinary fabrics, fully demonstrating the great potential of in MXene/WPU composite-coated fiber strain sensors wearable motion and healthcare monitoring.

**Supplementary Materials:** The Supplementary Materials can be found in the zip file. Figure S1. SEM images of (a)  $\text{Ti}_3\text{AlC}_2$ , (b) multi-layer  $\text{Ti}_3\text{C}_2\text{T}_x$ , and (c) few-layer  $\text{Ti}_3\text{C}_2\text{T}_x$  nanosheets. (d) XRD patterns of  $\text{Ti}_3\text{AlC}_2$  and  $\text{Ti}_3\text{C}_2\text{T}_x$ ; Figure S2. Solution photos after mixing MXene dispersion with (a) negatively-charged WPU, and (b) ordinary WPU

**Author Contributions:** conceptualization, Q.W.; methodology, J.C. Y.J. X.L. X.Y. J.Z. and Y.Z.; validation, J.C.; formal analysis, J.C. Y.J. X.L. J.Z. Y.Z. and Q.W.; investigation, J.C. Q.H. F.Y. G.L. and S.G.; resources, Y.J. X.L. X.Y.; data curation, J.C. and X.Y.; writing—original draft preparation, J. C.; writing—review and editing, Q.W.; visualization, J.C. Q.H.; supervision, X.Y. J.Z. Y.Z. and Q.W.; project administration, Q.W.; funding acquisition, Y.J. and Q.W.;

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