Microstructured capacitive sensor with broad detection range and long-term stability for human activity detection

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INTRODUCTION

With the increasing demand for artificial intelligence and the internet of things, the potential of flexible and wearable sensors in human–machine interaction, soft robots, and health monitoring applications have received great attention1–5. According to the sensing mechanism, the flexible sensors are divided into four types: resistive-type6–10, capacitive-type11–13, piezoelectric-type14–16, and triboelectric-type17–19. Among them, capacitive sensors have been intensively investigated owing to their characteristics of simple fabrication, good anti-interference, and excellent long-term stability20–22. However, their further applications are still restricted by the relatively low sensitivity and the sensing range23,24.

To date, researchers have made lots of efforts to improve the comprehensive performance of the capacitive sensor in various aspects, such as sensitivity, detection limit/range, response time, and stability25–29. For example, introducing the microstructure has been regarded as one of the effective strategies for remarkably improving the sensor sensitivity due to the distinct deformation of the microstructure and the subsequent increase in the capacitance variation. Ma et al. reported a capacitive pressure sensor consisting of two electrodes and wrinkled polydimethylsiloxane (PDMS) dielectric layer. Compared with the sensor without a microstructure, the fabricated sensor exhibited improved sensitivity from 0.01 to 2.04 kPa−1 in the range of 0–2 kPa24.

Additionally, bionic microstructures from natural plants, such as aureum leaf25, lotus leaf26, and rose petals27 also have attracted much attention because of their delicate and green/cost-effective features. Guo et al. proposed a capacitive sensor consisting of a dried flower petal or a leaf sandwiched between two flexible electrodes. The device worked within a broad-pressure range (0.6–115 kPa), the maximum sensitivity of which was 1.54 kPa−1. Furthermore, compared with single-component electrode materials, such as carbon nanotubes (CNTs)28,29, graphene30,31, conductive polymers32,33, metal nanostructures34–36, and MXene (Ti3C2Tx)37–40, the conducting composite has been widely utilized in the sensor electrode due to its combined advantages41–45. For example, the hybrid of one-dimensional (1D) and two-dimensional (2D) conducting materials could effectively prevent the aggregating and stacking of 2D material, thus forming a homogeneous connective network, which leads to good mechanical and electrical properties of the sensor electrode. Liu et al. reported a dendritic-lamellar MXene/carbon nanotube/polyvinylpyrrolidone electrode with high flexibility. The fabricated tactile sensor based on the composite electrode exhibited fast response, demonstrating for voice recognition and pulse measurement45.

However, the diverse applications require the pressure sensors to not only respond to a small stimulus but also work under an ultrahigh load46. For example, the huge impact force coming from a sudden striking usually exceeds hundreds of kilopascals, which is likely to cause the overload or failure of the sensors. Therefore, apart from high sensitivity and fast response, it is highly desirable to ensure a wide detection range and long-term stability of the device.

Herein, we reported a capacitive sensor whose overall performance was improved by designing the microstructure of the dielectric layer and selecting composite materials of the electrode. Inspired by the natural microstructures, we utilized the eggshell inner membrane (ESIM) as the template of the PDMS dielectric layer. Compared with other templates, the unique fibrous ESIM provided the nanoscale-template with a more elaborated microstructure47. We could achieve abundant micro-
fluctuations from the ESIM, which contributes to the remarkable deformation of the PDMS dielectric layer under the external stimuli. In addition, we proposed a composite electrode based on 2D MXene sheets and 1D Ag NWs. The Ag NWs can effectively decrease the restacking of MXene sheets, forming abundant conducting tunnels to facilitate electron transfer within the electrode. The fabricated MXene sheets and Ag NWs are closely contacted due to the electrostatic adsorption, which makes the conductive network have a stable structure. In this case, there is no relative slip between two conducting components even under high stress due to the tight combination, leading to a stable electron path within the conductive networks, which is beneficial to the long-term stability of the device under the repeated loading/unloading. As a result, the micro-structured sensor exhibited an improved sensitivity especially in the high-pressure range, broad detection range (0–600 kPa), low detection limit (<16 mg), the fast response time (in the millisecond range), long-term stability, and reproducibility.

RESULTS AND DISCUSSION

Micro-structured dielectric layer and composite electrode

To improve the detection range and long-term stability of the capacitive sensor, we used the micro-structured PDMS as the dielectric layer and MXene/Ag NWs as the rough electrodes. The microstructure of PDMS was replicated from the structure of the ESIM (Fig. 1a), and MXene sheets was prepared by etching the MAX phase and further delaminating (Fig. 1b, see Methods Section for details). The composite materials consisting of Ag NWs and MXene sheets within the electrode are expected to provide good contact and abundant conducting paths. The flexible capacitive pressure sensor was assembled using the micro-structured PDMS dielectric layer sandwiched between two MXene/Ag NWs electrodes (Fig. 1c).

Differently magnified SEM images disclose that the membrane is composed of fibrous protein with an average diameter of about 1 μm, presenting an interconnected network structure. There were a number of open spaces among the protein fibers (Fig. 2a, b and Fig. S1). The inverted microstructure was successfully transferred to the PDMS surface (Fig. 2c, d). These rugged inverse structures and the PDMS with small modulus could cause a wider range of compression deformation, which was expected to greatly improve the sensitivity and detection range of the sensor. The morphology and composition of electrode materials were further investigated. Figure 2e suggests that the MXene after etching features a multilayered structure. The element mapping illustrates that C, Ti, O, and F elements uniformly distribute across the multilayered MXene (Fig. 2f). As confirmed in the AFM image (Fig. 2g), MXene with a few layers was further acquired by ultrasonic treatment. The height distribution of MXene is about 5 nm, which is equal to 4–5 layers of Ti3C2Tx nanosheets stacked together (Fig. 2g, h). The obtained MXene sheets can be completely filtered to form an intact and smooth film due to the stacking (Fig. S2). The inset of Fig. S2 indicates that the MXene sheets uniformly disperse in water without obvious agglomeration. In addition, the average size of the MXene sheets is ~100 nm, which is similar to the diameter of the prepared Ag NWs (Figs. S3 and 2i). The 1D Ag NWs were tightly wrapped by the 2D MXene sheets after the combination of the two conductive materials (Fig. 2j), supplying an improved surface roughness of the electrode. XRD
characterization proves that we have successfully constructed MXene and metallic silver composites (Fig. 2k). Compared with the original material of Ti₃AlC₂, the (002) diffraction peak of MXene shifted to 5.9° after etching because of an increase in the layer spacing.

Due to the presence of the surface functional groups, the as-prepared MXene sheets were negatively charged with a zeta potential of −33.9 mV (Fig. 2l), while the surface of Ag NWs presented a positive charge with a zeta potential of +0.884 mV. Thus, the MXene nanosheets could closely adhere to the surface of Ag NWs owing to their strong electrostatic interaction, which is consistent with the result of SEM in Fig. 2j. The self-assembled composite (Fig. 5a) displayed a resultant zeta potential of −18 mV. In the composite system, 1D Ag NWs and 2D MXene sheets can be distributed across the surface of the flat sensor because of the planar and continuous contact interface between the electrode and dielectric layer (Fig. 3c). Therefore, the deformation and the contact area were hardly changed in this case. The device capacitance can be changed conveniently by controlling capacitance under various pressures by finite element analysis (FEA) (see Methods section for details). According to the definition of capacitance $C = \varepsilon_0 S/d$ (where $\varepsilon_0$ is the dielectric constant, $S$ is the area of the capacitor plate, and $d$ is the thickness of the dielectric layer or the distance between the two plates, $k$ represents the electrostatic constant, and $S$ is the area of the capacitor plate), the device capacitance can be changed conveniently by controlling $\varepsilon$, $S$, and $d$. To obtain a higher sensitivity of the sensor, the capacitance variation is expected to increase to the largest extent. Under a given load, the stress and deformation were uniformly distributed across the surface of the flat sensor because of the planar and continuous contact interface between the electrode and dielectric layer (Fig. 3c). Therefore, the deformation and the contact area were hardly changed in this case. The flat sensor exhibited high resistance to the external pressure, resulting in small structural compressibility, as well as few capacitance changes. When the deformation reached saturation, the capacitance turned to be a stable value, which is in accordance with the result in Fig. 3b. Thus, the sensing range of the flat sensor was limited to 100 kPa, characterized by low sensitivity and a relatively narrow sensing range.

The simulation result revealed that the micro-structured sensor experienced a higher level of deformation than the flat one under the same pressure due to the rough surface of the dielectric layer (Fig. 3c). Higher stress was concentrated on the bulges of the microstructure. As the external pressure exerted on the sensor increased, the contact area between the electrode and dielectric layer was gradually increased, while the distance between the two electrodes was decreased. Thus, the micro-structured sensor was able to present a larger capacitance variation under the same pressure, enabling higher sensitivity and a wider detectable range.

Sensing performance of capacitive sensors

We evaluated the performance of the micro-structured and flat capacitive sensors. The sensitivity of the sensor was measured through the slope of the relative change in the capacitance signal versus the applied pressure curve. The change rate of the capacitance of the flat sensor was ~50% under 100 kPa, presenting relatively a low sensitivity of 4.37 MPa⁻¹ (Fig. 3a). Furthermore, the change in the capacitance of the flat sensor was almost saturated over 100 kPa. Remarkably, the micro-structured sensor could work with a wider range of up to 600 kPa. The sensitivity value was increased to 10.13 MPa⁻¹ within 100 kPa, and especially by 54.3 and 95.7 times within high-pressure ranges of 100–400 kPa and 400–600 kPa (Fig. 3b).
respectively (Fig. 4b). It is also worth noting that measuring the
of the loading/unloading pressure indicated that the rise time and
response time and the detection limit. The time-response curve
performance parameters for the stress sensors involved the
of the sensor to rapid tapping using a
push-pull machine. Thus, the actual response time of the sensor
response time is limited by the movement rate (2.5 N/s) of the
instrument, and program computer (Fig. 4a), while the important

capacitance response within 50 ms (Fig. S7). A repeated signal can be
recognized during the loading and unloading stages (insets in Fig. 4g), verifying the excellent
mechanical stability of the sensor. To confirm the potential for
long-term application, the capacitance response of the sensor was
further measured after 150 days under the same measuring
condition (Fig. 4h, over 2700 cycles was shown in Fig. S8). The
sensor still exhibited good stability over the whole period, and the
capacitance variation was constant before and after long-term
storage and measurement due to the stable conductive network
within the electrodes. Furthermore, the SEM image of the dialectic
layer after the repeated loading reveals that the PDMS micro-
structure cannot be destroyed (Fig. S9).

**Demonstration of the micro-structured sensor**

Based on the above characterizations and analyses, the flexible
capacitive sensor demonstrated enhanced sensitivity, a wide
detection range, low detection limit, rapid response, and long-
term stability. Moreover, it was envisioned that the micro-
structured sensor could have outstanding practicability when
detecting diversified body movements. To demonstrate this, the
sensor was firmly attached to various body parts of a healthy 26
years old volunteer to collect the capacitance signals in response
to the body movements (Fig. 5a). The regular and repetitive
output of the electrical signal could be observed when the

**Fig. 3** Comparison of the flat and micro-structured sensors. a Capacitance changes of the flat sensor under the external pressure within 100 kPa; b Sensitivity comparison of the flat and micro-structured sensors within the range of 0–600 kPa; c Schematic diagram of the two sensors under the external pressure from Comsol simulation; d Compression displacement comparison of the two sensors; e Schematic diagram of the changes in the two sensors’ distance and contact area under a given pressure.

In addition, the result of the simulated displacement between the
two electrodes under various pressures and the structural changes
of the capacitive sensors further proved that a larger deformation
occurred under the same load in the case of the micro-structured
sensor (Fig. 3d, e).

Therefore, the improved performance of the micro-structured
sensor can be attributed to the following three aspects. First, the
microstructure enables the sensor to experience a higher level of
deformation under the same pressure, thus generating more
obvious changes in the distance between the two electrodes, as
well as the capacitance variations. Second, the high roughness of
the electrode due to the combination of 1D and 2D conductive
materials provides the improvement of the void space within the
electrode. Thus, under a given loading, the stress sensor based on
the MXene/Ag NWs electrode is expected to exhibit a large
change in the $d$ and $C$, and then generating a higher sensitivity
coefficient (Fig. S6 shows the capacitance changes of the sensor
based on smooth ITO/PET electrodes and the micro-structured
PDMS dielectric layer, revealing a lower sensitivity within whole
working range). Third, there is an air gap between each unit of the
bulges in the microstructure. When the load was enhanced, the air
gap would be gradually filled by PDMS material. Accordingly, the
comprehensive dielectric constant $\varepsilon$ of the dielectric layer rose
($\varepsilon_{\text{Air}} = 1, \varepsilon_{\text{PDMS}} = 2.5–2.7$), causing a larger capacitance change.

The instruments for testing the flexible pressure sensors
included the push-pull machine, LCR capacitance measurement
instrument, and program computer (Fig. 4a), while the important
performance parameters for the stress sensors involved the
response time and the detection limit. The time-response curve
of the loading/unloading pressure indicated that the rise time and
fall time of the micro-structured sensor was 280 and 290 ms,
respectively (Fig. 4b). It is also worth noting that measuring the
response time is limited by the movement rate (2.5 N/s) of the
push-pull machine. Thus, the actual response time of the sensor
could be less than 50 ms. We also tested the capacitance response
of the sensor to rapid tapping using a finger, proving that the fast
response within 50 ms (Fig. S7). A repeated signal can be
recognized during the loading and unloading process of a grain
of rice with a weight of 16 mg (Fig. 4c), denoting that the sensor
can be used to reliably detect a subtle stimulus by the capacitive
response. Additionally, the sensor showed stable signals in single
and multiple cycles under the pressures of 10, 20, 35, and 45 kPa,
respectively (Fig. 4d, e). The response to each loading exhibited
stepped waves and stable square wave-forms, revealing excellent
sensing stability of the sensor within a wide pressure range.
Moreover, the micro-structured sensor also demonstrated a
precise response independent of the loading speed (0.10, 0.25,
and 2.50 N/s) under the application of 2.5 N loading/unloading
cycles (Fig. 4f). The rapid change and good recovery of the
capacitance at each speed evidenced the fast response and
excellent reversibility of the sensor.

To assess the reproducible sensing performance of the device,
the sensor was loaded and unloaded with a constant pressure of
40 kPa dynamically for over 20,000 s at least (Fig. 4g). The micro-
structured sensor exhibited remarkable robustness as the
capacitance could recover its initial value even after being
subjected to 1800 cycles. The magnified parts clearly showed no
obvious amplitude change in the capacitance value at different
loading/unloading stages (insets in Fig. 4g), verifying the excellent
mechanical stability of the sensor. To confirm the potential for
long-term application, the capacitance response of the sensor was
further measured after 150 days under the same measuring
condition (Fig. 4h, over 2700 cycles was shown in Fig. S8). The
sensor still exhibited good stability over the whole period, and the
capacitance variation was constant before and after long-term
storage and measurement due to the stable conductive network
within the electrodes. Furthermore, the SEM image of the dialectic
layer after the repeated loading reveals that the PDMS micro-
structure cannot be destroyed (Fig. 59).
volunteer repeatedly pronounced the word “studying” (Fig. 5b), caused by the neck muscle movement and the vocal cord vibration. When the sensor was attached to the cheek, the stress of the air blowing on the device were distinctly responded (Fig. 5c). Furthermore, the stable capacitance change-time curves were obtained when the volunteer clenched and released his fist attached by the micro-structured sensor (Fig. 5d). The sensor was also capable of monitoring other body movements, such as the bending of the finger, elbow, and knee. The favorable reversibility during each testing cycle was achieved (Fig. 5e, f, g). Additionally, the angle recognition of the sensor was also demonstrated by attaching the sensor to the index finger (Fig. S10). When the sensor was placed on the sole, the highly stable signal output illustrated that the sensor could be used to monitor the response to walking (Fig. 5h). As a result, the sensor’s function of efficiently detecting diverse body movements within a broad detection range was proven.

The sensor was fixed on the surfaces of different objects to measure the sensing capability of the device as a tactile sensor in specific activities (Fig. 6). The stable electrical signals responding to the repeated quick touching and weight pressing on the sensor reflected the rapid response and good mechanical stability of the sensor (Fig. 6a, b). The sensor was also adhered to the beaker and keyboard to monitor the real-time grasping and knocking, which indicated the stable wave-forms and constant capacitance variations in response to the repeated actions (Fig. 6c, d). Figure 6e displays the signal output of the pressure sensor in response to a noncontact force from air blowing of aurilave. The sharp and

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**Fig. 4.** Electron-mechanical characteristics of the flexible capacitance sensor. a Schematic diagram of the testing platform; b Response time of the sensor with the insets corresponding to the loading and unloading process; c Detection limit test by loading/unloading a grain of rice with a weight of 16 mg; d Capacitance change with the gradient increase and decrease under 10–45 kPa; e Cyclic capacitance changes under 10–45 kPa at the same frequency; f Ten cycles of the sensor with different loading speeds; g, h Stability test of the sensor under 40 kPa before and after 150 days; the insets are partially magnified curves for different cycle stages.
repeated wave-forms reflected the high sensitivity and fast response of the sensor when detecting the real-time wind pressure. To assess the detection capability under high pressure, the six independent sensors were fabricated into an array to evaluate the volunteer’s foot pressure distribution (Fig. 6f). The distinct capacitance values indicated that the sensor array could distinguish different parts of foot pressure when the volunteer was standing. Strike tests of the sensor at different pressures disclosed that the device rapidly responded to the repeatedly strong strikes by the fingertip, stainless cone, and pen point (Fig. 6g–i).

These results demonstrated the outstanding sensing ability of the micro-structured sensor to monitor diverse human activities and physical stimuli, accompanied by the characteristics of high sensitivity, fast response, excellent stability, and broad detection range. The synergy of the rough surface of the conductive materials and the nanoscale-microstructure of the dielectric layer contributed to the improvement of the sensor performance. The proposed sensor shows good prospects in healthcare, soft robots, human–machine interaction, and wearable devices.

METHODS

Synthesis of Ag NWs and MXene

A hydrothermal method was used for preparing Ag NWs according to our previous work6,57. First, 0.788 g silver nitrate, 0.53 g glucose, and 0.23 g ferric sulfate were dissolved in 100 mL distilled water to obtain a light-yellow solution. Second, 4.5 g poly(vinyl pyrrolidone) (K30) was added to the above solution under magnetic stirring for 2 h. Third, the homogeneous mixture was transferred to a Teflon autoclave of 100 mL, and then heat-treated at 180°C for 9 h. Finally, the gray-green Ag NWs were collected by repeated centrifugation and then dispersed in ethanol. Ti₃C₂Tₓ was synthesized by a selective Al etching of MAX phase precursor powder (Ti₃AlC₂, 11 Technology Co., Ltd., China) by a minimally intensive layer delamination method. First of all, 1.6 g LiF was dissolved in 9 M HCl of 20 mL, and then 1 g Ti₃AlC₂ powder was added to LiF/HCl solution with the assistance of magnetic stirring to avoid a sharp increase in the reaction temperature. The etching reaction occurred at 35 °C for 48 h with the persistent stir. Subsequently, deionised water was used for washing the etched product until the pH value of the supernatant reached 6. Finally, the Ti₃C₂Tₓ dispersion was ultra-sonicated for 2 h under the condition of the ice bath and Ar atmosphere. The delaminated Ti₃C₂Tₓ flakes were collected from the upper solution.

Fabrication of micro-structured PDMS dielectric layer

The microstructure of the PDMS (Sylgard 184, Dow Corning) dielectric layer was replicated from the ESIM. First, the ESIM was peeled and then cleaned with deionised water. Second, the mixture of PDMS pre-polymer and a curing agent (10:1 by weight) was firstly vacuum-filtrated on a filter membrane, and then we poured the mixture of PDMS pre-polymer and a curing agent (10:1 by weight) onto the MXene/Ag NWs-filter membrane. After cured at a
temperature of 60 °C for 4 h, the composite conducting materials are embedded into the PDMS layer. The MXene/Ag NWs was completely transferred to the PDMS surface by removing the filter membrane, acquiring the electrodes. Finally, the electrodes and dielectric layer were cut with an area of 10 mm × 10 mm and then integrated into a sandwiched device. For performance comparison, we fabricated a flat sensor by using the same MXene/Ag NWs composite electrodes and a flat PDMS dielectric layer.

Characterization

A field emission scanning electron microscope (FE-SEM, Zeiss Sigma 500, Germany) was used for observing the morphology of the dielectric layer and electrode materials. The composition of the conductive materials was characterized through an X-ray diffractometer (XRD, X’pert Pro MFD, Panalytical, The Netherlands) with a Cu Kα radiation (λ = 1.54178 Å). The thickness of MXene sheets was determined by an atomic force microscope (AFM, Multimode 8, Bruker, Germany). The surface potential of the conductive materials was tested using a Malvern Zetasizer (Nano ZS, England). The stress was applied to the sensors by a push-pull force gauge (LE, Lishi, China). The response performance of the stress-dependent sensor was evaluated by measuring the real-time capacitance changes of the device by an LCR (Inductance, Capacitance, Resistance) measurement instrument (IM3526, Rizhi, Japan). The capacitance of the sensor was tested under the frequency of 500 kHz.

In order to more accurately evaluate the advantage of the micro-structure in the flexible capacitive sensor, the FEA was conducted to compare the two models based on the micro-structured and planar PDMS dielectric layer by a commercial package Comsol 5.4. The micro-structured model was constructed according to the actual replicated surface of the dielectric layer with the electrodes replaced by the metal plates.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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AUTHOR CONTRIBUTIONS
X.H. and G.C. designed this work and analyzed the data; Z.L., G.S., and X.H. fabricated, characterized the electrodes and devices; J.L., Y.Z., T.L., and J.H. discussed the results; Y.X., C.Z., and D.Y. involved in the analysis of characterization; X.H. and Z.L. wrote the manuscript; G.C. revised the manuscript. All authors read and approved the final manuscript.

COMPETING INTERESTS
The authors declare no competing interests.

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