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Flexible and transparent capacitive pressure sensor with patterned microstructured composite rubber dielectric for wearable touch keyboard application

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ABSTRACT The development of pressure sensors with highly sensitivity, fast response and facile fabrication technique is desirable for wearable electronics. Here, we successfully fabricated a flexible transparent capacitive pressure sensor based on patterned microstructured silver nanowires (AgNWs)/polydimethylsiloxane (PDMS) composite dielectrics. Compared with the pure PDMS dielectric layer with planar structures, the patterned microstructured sensor exhibits a higher sensitivity (0.831 kPa^{-1}, <0.5 kPa), a lower detection limit, good stability and durability. The enhanced sensing mechanism about the conductive filler content and the patterned microstructures has also been discussed. A 5×5 sensor array was then fabricated to be used as flexible and transparent wearable touch keyboards systems. The fabricated pressure sensor has great potential in the future electronic skin area.

Keywords: silver nanowires, capacitive pressure sensor, flexible, transparent, wearable electronics, e-skin

INTRODUCTION

Flexible and bendable pressure sensors with high sensing performances have obtained great attention [1–6], due to their broad applications in human-machine interactions [5,7,8], health monitoring [9,10], medical diagnostics [2,11,12] and artificial intelligence [13], etc. Recently, high sensitivity, large-scale and high resolution pressure sensor have made significant breakthrough based on different physical transduction mechanisms, such as piezoelectricity, piezoresistivity and capacitance. Compared with the other types of pressure sensors, capacitive pressure sensors with high accuracy in detecting static loads, low power consumption, low hysteresis and large response ranges, have obtained great success in the field of consumer electronics, including biometric identification [13,14], touchpads [7,15] and touchscreens.

Due to the transduction mechanism to the applied mechanical force, the sensitivity of the capacitive pressure sensor primarily depends on the distance between the two electrodes and the dielectric constant of the dielectric layer. Therefore, capacitive sensors require a dielectric material that is easy to compress and responds rapidly to pressure applied and released. In addition to using foam as dielectric material to detect moderate pressure, elastomers are also incorporated into capacitive pressure sensors. However, the inherent characteristics of a single dielectric limit the further development of capacitive pressure sensors. Therefore, to increase the sensing performance, some effective methods have been investigated, including doping fillers in insulating elastic dielectrics [16–20], introducing the ordered microstructures to the dielectric [14,20–24], changing the internal microstructure of the dielectric [15,25], and so on. For example, Schwartz *et al.* [26] reported a flexible capacitive pressure sensor embedded capacitive sensing element with a microstructured elastomer layer which revealed a fast response within a millisecond range and a great mechanical flexibility. However, few reports focus on the synergistic effect of the fillers and microstructures to improve the capacitive sensing performance.

Herein, we present a highly sensitive, flexible and transparent capacitive pressure sensor with the silver nanowires (AgNWs)/polydimethylsiloxane (PDMS) composite dielectric films. To further enhance the sensitivity, patterned pyramid-type microstructures were introduced to the composite film by using a simple and low-cost method. With patterned microstructures and sensitive composite dielectric films, the sensors have high sensitivity in the low voltage range (0.831 kPa^{-1}, <0.5 kPa)

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and very low detection limits of 1.4 Pa, as well as ultrafast response times. It also exhibits excellent operational durability and repeatability. In addition, we fabricated a flexible sensor array for wearing flexible touch keyboard, demonstrating that this capacitive pressure sensor has great potential in the application of human-computer interaction and electronic skin.

EXPERIMENTAL SECTION

Fabrication of micropatterned silicon mould

200 nm SiO_2 was deposited on a clean (100) Si wafer using plasma enhanced chemical vapor deposition (PECVD). Then the SiO_2 oxide layer was photolithographed using a mask plate. The exposed SiO_2 patterns was etched by buffered oxide etch (BOE) (NH_4F:HF=6:1, *v*/*v*). Next, the substrate was wet etched anisotropically using tetramethylammonium hydroxide (TMAH) solution (20 wt% TMAH:isopropanol=5:1, *v*/*v*) at 80°C under stirring for 60 min. The remaining SiO_2 layer was removed by BOE. After washing with deionized (DI) water, the Si master was deposited on 50 nm Au to prevent adhesion.

Synthesis process of AgNWs

AgNWs were synthesized by hydrothermal method. 0.24 g Polyvinylpyrrolidone (PVP) was fully dissolved in 30 mL ethylene glycol under 65°C for 2 h. After the solution was cooled to room temperature, 0.5 g of $AgNO_3$ was added and stirred until completely dissolved. 7 g $FeCl_3$ solution (600×10^{-6} mol L^{-1}, ethylene glycol) was poured into the above solution and stirred for 3 min. The mixed solution was then immediately transferred to reaction kettle and placed in an oven at 130°C for 5 h. Finally, the precipitate was centrifuged at 7,500 rpm for 5 min with ethylene glycol and then centrifuged twice with acetone and ethanol at 3,500 rpm for 5 min, respectively. The obtained AgNWs were dispersed in ethanol at a concentration of 0.3 mg mL^{-1}.

Fabrication of AgNWs/PDMS mixture solution and the micro-structured composite dielectric layers

First, the PDMS elastomer (Sylgard 184, Dow Corning) and the curing agent were mixed at a mass ratio of 10:1 and stirred for 30 min, and then evacuated for 15 min. The prepared AgNWs were dispersed in ethanol and its concentration was 0.3 mg mL^{-1}.

Thereafter, different proportions of the AgNWs solution were added to 1 g PDMS mixture and stirred fully with a magnetic bar to obtain the composite solution with different doping ratio. In order to remove ethanol, the resulting solution was centrifuged at 8,000 rpm for 5 min and removed the supernatant and then the composite solution was stirred for 20 min and evacuated at 50°C for 15 min. Finally, it was spin-coated onto the micropatterned Si mould at 1,000 rpm and cured at 80°C for 2 h before being peeled off.

Characterization

The scanning electron microscopy (SEM) images were characterized using an FESEM Shimadzu Japan (SSX-550). The optical images were captured using a Canon camera. The computer controlled movable stage (Shandu SJY-500V) and force gauge (Shandu SP-5 and SH-500B) were used to apply the static pressure. A home-made force supplied equipment was used to apply dynamic pressure measurements. The electrical characterization of the flexible capacitive pressure sensor was measured with Keithley 4200 under the signal of 1 V at 2 MHz .

Materials

All the reagents including ethylene glycol (anhydrous, 99.8%, Aldrich), PVP (M_w = 360,000, Aldrich), ferric chloride (Aldrich), silver nitrate (Aldrich), acetone (AR, 99.5%), and anhydrous ethanol (AR, 99.5%), were used without further purification. PDMS used was SYLGARD 184 Silicone Elastomer Base and Curing Agent. The indium tin oxides (ITO)/polyethylene terephthalate (PET) film (50 μm) was commercially available.

RESULTS AND DISCUSSION

The schematic illustrations for the fabrication of micropatterned Si mould and flexible microstructured composite AgNWs/PDMS film were illustrated in Fig. 1. The detailed process of fabrication was described in the experimental section. Briefly, the micropatterned Si mould was first fabricated using conventional photolithography and wet etching (Fig. 1a). The microstructures of the Si mould were regular and uniform as shown in Fig. S1. Moreover, the mould can be reused for many times, thus the repeatedly operations do not affect the uniformity of the transferred microstructured dielectric films. The prepared AgNWs/PDMS solution was spin-coated on the Si mould, which were then peeled off to obtain the flexible microstructured AgNWs/PDMS composite dielectric film (Fig. 1b). The concentration of AgNWs in ethanol and the detailed fabrication method of AgNWs/PDMS mixture solution can be find in the experimental section. Then we used the as-obtained film to prepare a flexible capacitive pressure sensor.

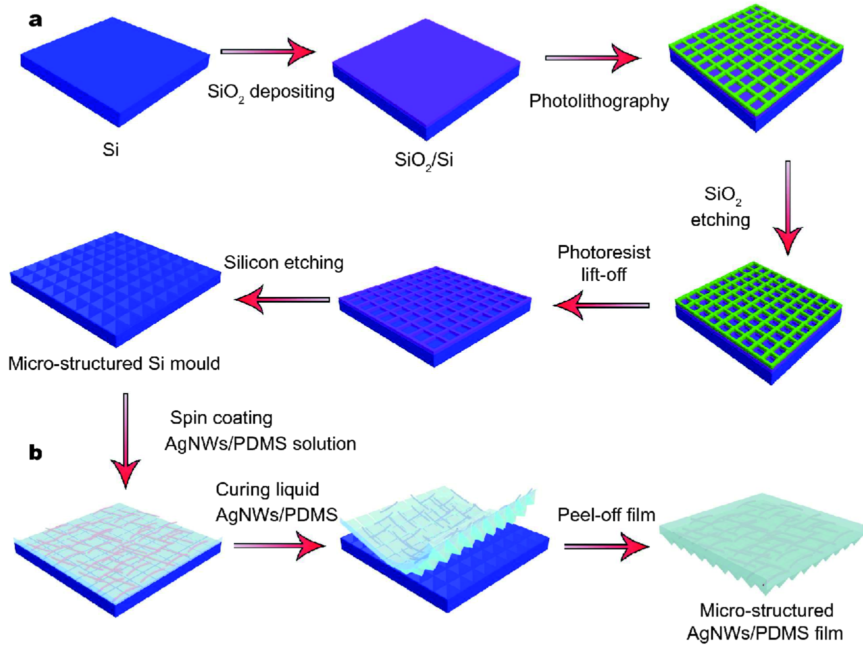

Figure 1 Schematic illustration for the fabrication of (a) micropatterned Si mould and (b) flexible microstructured AgNWs/PDMS composite dielectric film.

Fig. 2a illustrates the device structure of the flexible capacitive pressure sensor using AgNWs/PDMS composite films as the dielectric films in the middle, sandwiched by two ITO/PET (~50 μm) films acting as the upper and lower electrodes. As the PET and AgNWs/PDMS films are flexible, the as-made devices are bendable and hence wearable. The optical image of a single pressure sensor device shows that the device has good transparency (Fig. 2b). The transparency of the composite films can be easily regulated by doping different mass ratio of AgNWs. SEM image shows the as-grown AgNWs with an average length of 150 μm and diameter of 100 nm (Fig. S2). As shown in Fig. 2c, d, the pyramid-shaped microstructures were regularly arranged and had the same dimensions with a length and a height of about 11.5 μm and 7.6 μm, respectively. The thickness of the micro-structured dielectric layer is about 98.8 μm, as shown in Fig. S3. Fig. 2e shows the optical transmittance of the AgNWs/PDMS films with various mixing ratios of AgNWs. When the mass ratio of AgNWs and PDMS is 0%, 0.03%, 0.06% and 0.12%, the transmittance of the AgNWs/PDMS film is 98, 91, 87 and 71% under 550 nm light illumination, respectively.

The sensing mechanism of the capacitive pressure sensor based on the AgNWs/PDMS dielectric layer was illustrated in Fig. 3a. The basic sensing mechanism of the traditional capacitive pressure sensing is equivalent to the parallel plate capacitor, and the capacitance value C is as follows: $C=\varepsilon_0\varepsilon S/d$ (Equation 1), where ε_0 is the vacuum dielectric constant and ε is the dielectric constant of the composite film, and S is the relative plate area, and d is the distance between the plates. When the sensor is not loaded, the distance between the upper and lower electrodes is fixed leading to a certain initial capacitance value. However, when the force is applied to the pressure sensor, the dielectric is deformed, and the distance between the plates is reduced to d. As a result, the capacitance value C becomes large. Thus, a conventional capacitive pressure sensor may provide a significant change in capacitance proportional to the applied pressure.

In addition to the distance d, the change in the dielectric constant ε can also cause a change in the capacitance value C. Therefore, we controlled the dielectric constant of the dielectric layer by doping different proportions of AgNWs in the PDMS dielectric to improve the sensitivity and response range. The change of dielectric constant of the AgNWs/PDMS composites under pressure can be interpreted by the Kirkpatrick and Zallen statistical percolation model [18,29–32], which is used to predict the electrical properties of a percolation system with non-interacting randomly dispersed fillers. Here, the capacitance value C is as follows: $C=\varepsilon_0\varepsilon_a S/d$ (Equation 2) and $\varepsilon_a=\varepsilon_d(f_c-f)^{-S}$, $f_c>f$ (Equation 3), where ε_d is the effective

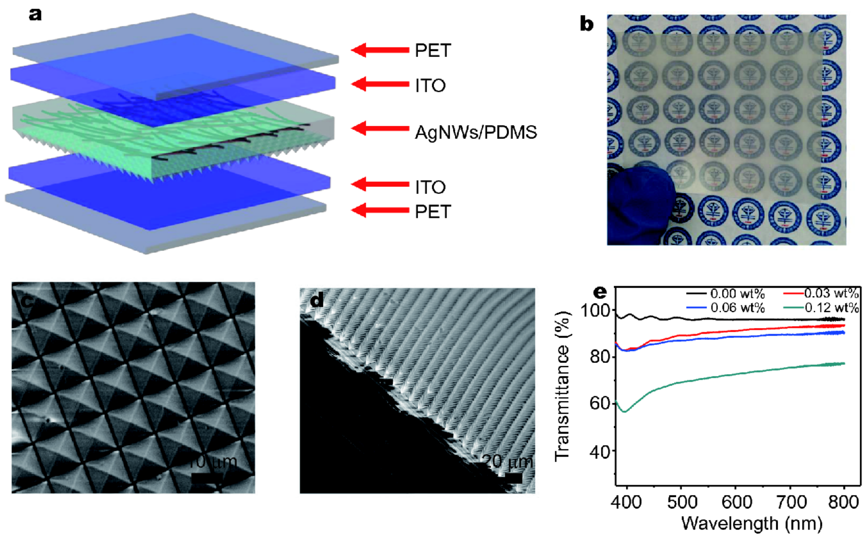

Figure 2 (a) Architecture of the flexible capacitive pressure sensor. (b) The photo image of a single pressure sensor device. (c) Top and (d) tilted SEM images of the micro-structured AgNWs/PDMS film. (e) Optical transmittance of the AgNWs/PDMS films with various mixing ratios of AgNWs.

dielectric constant of the composite dielectric film, and the value of ε_a can be determined by the content of the filler material and the deformation of the dielectric under pressure. f_c, f and ε_d are the percolation threshold, the filling factor, and the conductivities of the dielectric phases, respectively. According to the Equations (2) and (3), the incorporation of AgNWs can change ε_a of PDMS dielectrics, and the change of ε_a will be maximized when the concentration of AgNWs reaches a certain content. Combined with the reduced distance d caused by pressure, the sensitivity of the sensor can be further enhanced.

First, to optimize the doping ratio of AgNWs, the response performance of the pressure sensors has been characterized. Fig. 3b shows the capacitance changes of the non-patterned sensors under pressure with various mixing ratios of AgNWs. The sensitivity S of the pressure sensor can be defined as: $S=((C-C_0)/C_0)/\Delta p$, (Equation 4), where p is the applied pressure, C and C_0 represent the capacitance with and without pressure. The sensitivity of the sensor based on the unstructured PDMS film without AgNWs was much lower than that with the AgNWs/PDMS composite film. The results show that the sensor with 0.12 wt% AgNWs exhibits the highest sensitivity. In the pressure range of 1 kPa, the sensitivity of the AgNWs/PDMS composite film (0.059 kPa^{-1}) is about five times higher than that of the pure PDMS film (0.012 kPa^{-1}). And thus, the doping ratio of 0.12 wt% was chosen as the optimal dielectric of the capacitive pressure sensor.

On this basis, in order to further improve the sensitivity of the sensor, the pyramid-shaped microstructure was then introduced to the composite film. Fig. 3c, d show the sensitivity curves of the pressure sensor based on different types of dielectric films. It can be seen clearly that the sensor based on microstructured AgNWs/PDMS dielectric films with 0.12 wt% AgNWs exhibits higher pressure sensitivity than that based on the non-patterned or microstructured PDMS dielectric layer without AgNWs. For the sensor with 0.12 wt% AgNWs, the sensitivity is as high as 0.831 kPa^{-1} in the low pressure range. Although the value decreases (0.063 kPa^{-1}) as the pressure increases, it is also comparable to some reported capacitive pressure sensors [11,14]. The outstanding sensitivity of the sensor at low pressure range combined with wide sensing range make it possible to detect slight touch information. Similarly, the sensitivity of the sensor based on microstructured PDMS dielectric layer without AgNWs is about 0.218 kPa^{-1} at low pressure and decreases to 0.035 kPa^{-1} with increased pressure. Compared with the previous researches shown in Fig. 3e and Table S1, the as-prepared sensors have better performance such as high sensitivity and low detection limit. These results show that the sensing performance of the as-fabricated capacitive pressure sensor based on a conventional PDMS film is successfully enhanced by changing geometrical structures and additional factors such as dielectric constant ε.

According to the above results, here we just discuss the pressure sensor based on microstructured 0.12 wt% AgNWs/PDMS dielectric film. The reliable capacitance changes of the sensor under different pressures are shown

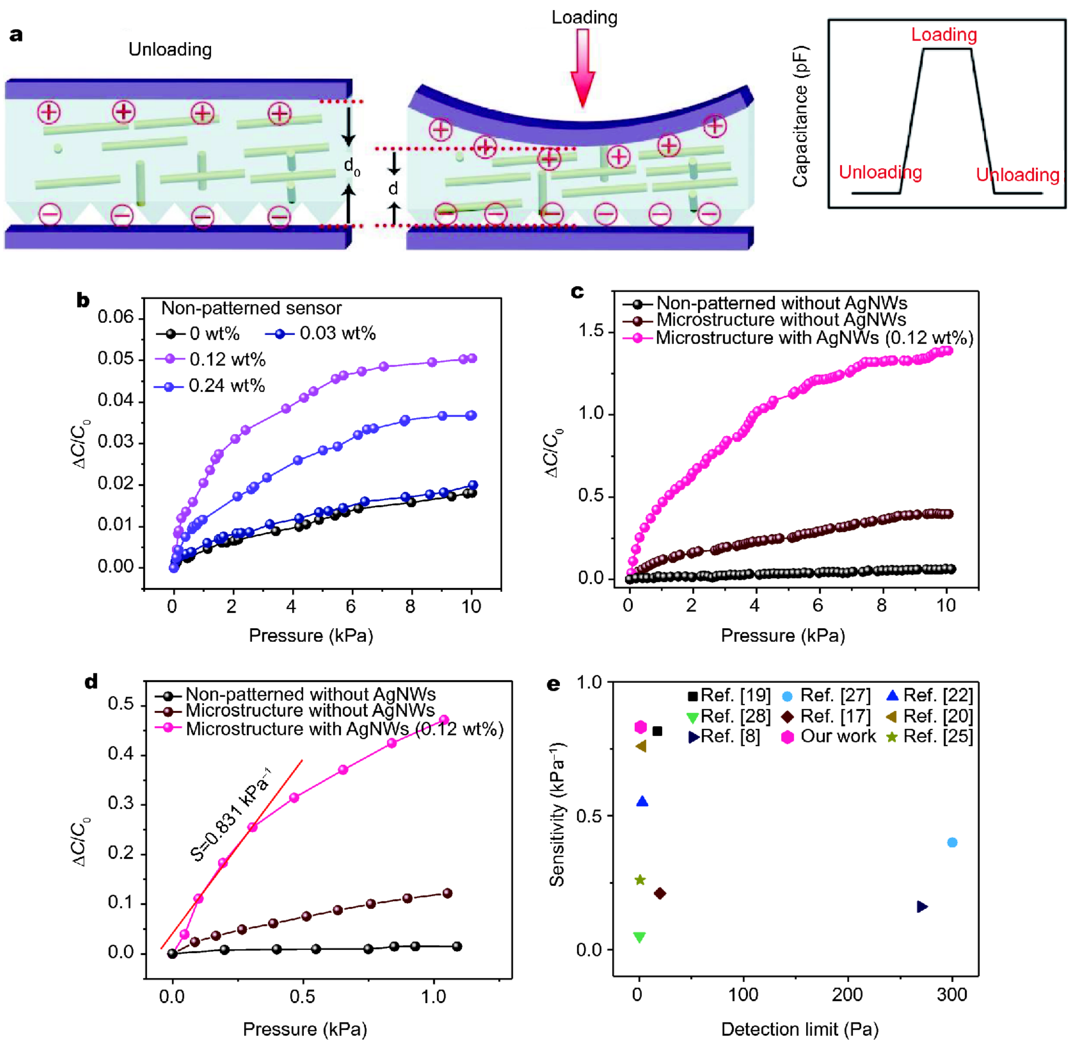

Figure 3 (a) Schematic illustration of the sensing mechanism of the capacitive pressure sensor. (b) Capacitance changes of the non-patterned sensors under pressure with various mixing ratios of AgNWs. (c, d) Sensitivity curves of the pressure sensor with different types of dielectric layer under applied pressure. The micro-structured sensor based on AgNWs/PDMS dielectric layer with 0.12 wt% AgNWs exhibits higher pressure sensitivity than that based on the non-patterned or micro-structured PDMS dielectric layer without AgNWs. (e) Comparison of the sensing performance of our work and previous research results [8,17,19,20,22,25,27,28].

in Fig. 4a. It can be seen that the sensors exhibit a very stable response under dynamic pressure, which can be stably operated under static pressure and accurately measure the applied force. Fig. 4b shows the real-time response curves of both types of microstructured sensors under pressures of 0.1 kPa and 0.2 kPa, respectively. The results show that the microstructured pressure sensor with 0.12 wt% AgNWs exhibits a higher sensitivity and reproducibility than the sensors without AgNWs. Fig. 4c and insets show the fast response and relaxation times of the pressure sensor. When the pressure was loaded to the pressure sensor, the response time was less than 30 ms, and a short relaxation time (less than 60 ms) was obtained when the pressure was unloaded from the sensor. This pressure sensor based on AgNWs/PDMS composite film can also detect small changes in pressure. Due to the high sensitivity and fast reaction time of the pressure senor, some ultra-light objects such as a small piece of paper (89 mg, 1.4 Pa), etc. loaded onto the sensor can induce the capacitance changes as shown in Fig. 4d. Durability and reliability are two key factors in the practical application of pressure sensors. Hence, the cyclic stability of the pressure sensor was then measured. Fig. 4e shows the capacitance change curves under a pressure of 1 kPa. It is worth noting that even after 10,000 cycles, the attenuation of the capacitance change can be negligible.

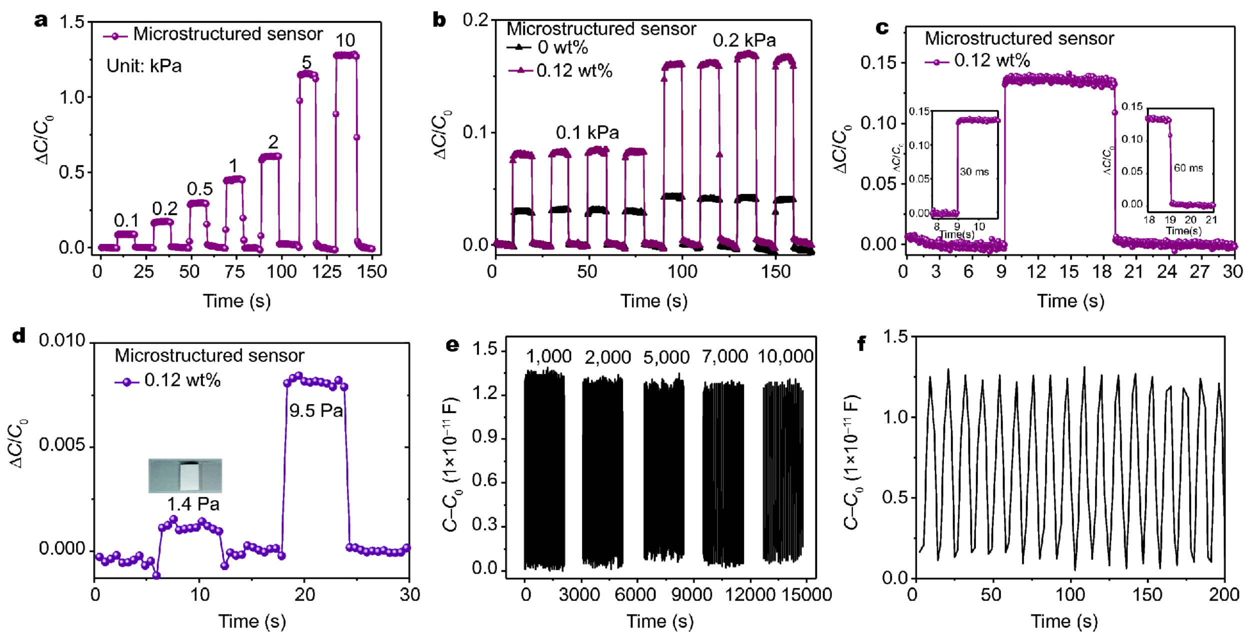

Figure 4 Characterization of capacitive pressure response of the sensor with the microstructured 0.12 wt% AgNWs/PDMS dielectric layer. (a) Reliable capacitance change of the sensor under different pressures. (b) Repeated real-time response curves of both types of micro-structured sensors under pressures of 0.1 kPa and 0.2 kPa, respectively. (c) Fast response time (<30 ms) and relaxation time of the sensor. (d) Transient response to the placing and removal of a small piece of paper, the first corresponding to a pressure of only 1.4 Pa. (e) Capacitance change curves recorded after 1,000, 2,000, 5,000, 7,000 and 10,000 cycles, respectively under a pressure of 1 kPa. (f) Magnified view of (e) after 7,000 loading-unloading cycles.

The entire 10,000 cycles are shown in Fig. S4. The large number of repetitive cycles confirms the excellent reproducibility of the sensor. As shown in the enlarged view (Fig. 4f), almost the same sharp peaks were observed in each cycle. The results show that the pressure sensors based on AgNWs/PDMS composite film have a long working life and stability.

In order to realize the practical application of flexible capacitive pressure sensor in human-computer interaction and electronic skin, it is necessary to fabricate a large-scale flexible pressure sensor array. Fig. 5a shows the schematic illustration of the final flexible 5×5 e-skin device and an enlarged pixel with a sandwich structure. By detecting the capacitance variation of each pixel, the array device can be used as a flexible wearable keyboard to implement the function of a conventional keyboard. The system-level block diagram in Fig. 5b shows the signal switching, conditioning, processing and wireless transmission paths from the signal conditioning of each pixel and output the corresponding conversion signal. The oscillating circuit is used to detect the input capacitance change of the array device. In addition, the microprocessor determines the key information by calculating and detecting the frequency of the oscillation circuit, and controls the input switching circuit to acquire the signals of the respective pixel points. Finally, the data is wirelessly transferred to the phone application *via* the WiFi module. Fig. 5c shows the smart keyboard that fits into the skin and mobile phone software. The flexible sensor array is able to achieve the function of keyboard. Demonstrations such as real-time inputting word "f" (Fig. 5d) and "flextronics" (Fig. 5e) are shown in the mobile application. Remarkably, such matrix of pressure sensor array reveals a great potential in the applications of human-computer interaction.

CONCLUSIONS

In summary, we used a simple and low-cost process to fabricate flexible, transparent and ultrasensitive capacitive pressure sensors by introducing patterned microstructured AgNWs/PMDS composite dielectric films. The as-fabricated capacitive pressure sensor has a large capacitive response (0.831 kPa^{-1}) when different pressures are applied, and can easily detect minute pressure (1.4 Pa). Moreover, the capacitive pressure sensor has a fast response (<30 ms) and relaxation (<60 ms) time. It also exhibits a high stability and durability after repeated cycles of more than 10,000 times. We also demonstrated the expansion of our sensors in the field of wearable electronic skin by making a 5×5 array of wearable flexible

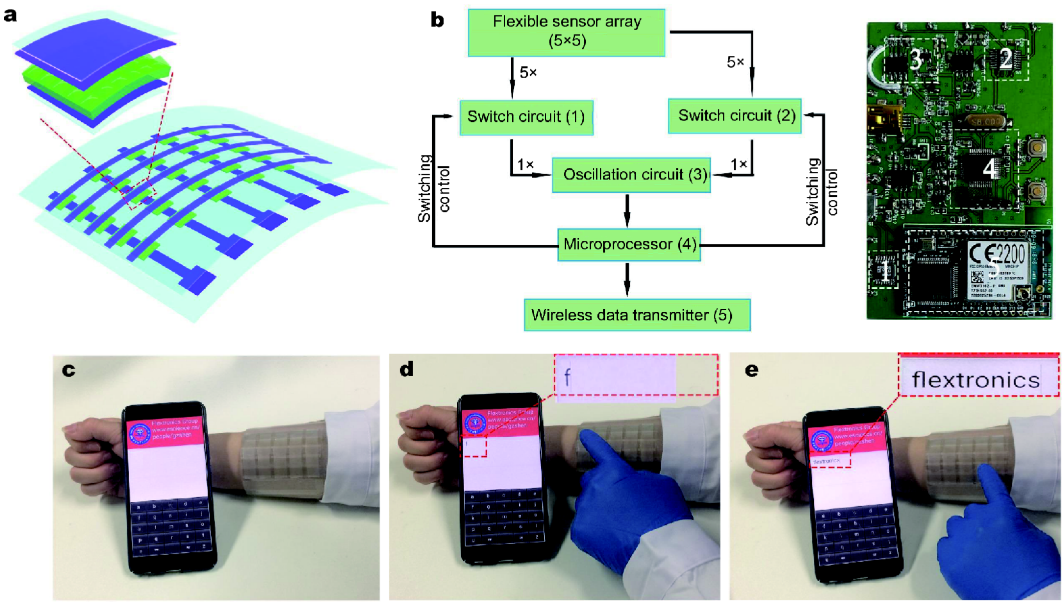

Figure 5 (a) Schematic illustration of the final flexible e-skin device and an enlarged pixel with a sandwich structure. (b) System-level block diagram of the wireless printed circuit board (PCB) showing the signal switching, conditioning, processing and wireless transmission paths from sensors to the mobile application (numbers in parentheses indicate the corresponding labelled components in the photo of the wireless PCB). The flexible sensor array is able to achieve the function of the keyboard (c). Demonstrations such as real-time inputting word "f" (d) and "flextronics" (e) are shown in the mobile application.

keyboards. The array-wearable keyboard shows a high sensitivity which can clearly distinguish the applied pressure to each pixel and implements a function similar to that of a conventional keyboard.

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Author contributions Shi R and Lou Z designed the devices and experiments; Shi R performed the experiments; Shi R, Lou Z and Chen S analyzed the data; Shi R and Chen S synthesized the Ag nanowires; Shi R wrote the paper with support from Lou Z and Chen S. All authors contributed to the general discussion.

Conflict of interest The authors declare no conflict of interest.

Supplementary information Supporting data are available in the online version of the paper.

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基于银纳米线/PDMS微结构复合电介质的柔性透明电容式压力传感器及其在穿戴式触摸键盘的应用

史瑞龙[1,2], 娄正[1*], 陈帅[1], 沈国震[1,2*]

摘要 对可穿戴电子设备来说，采用简易制备技术得到具有高灵敏度、快速响应的压力传感器至关重要．本文成功地研制了一种基于特殊微结构银纳米线/PDMS复合电介质层材料的柔性透明电容式压力传感器．与采用纯PDMS平面结构的电介质层器件相比，有微结构的传感器具有更高的灵敏度(0.831 kPa^{-1}, <0.5 kPa)，更低的检测范围，更好的稳定性和耐久性．本文对导电填料含量和微结构的增强传感机理也进行了讨论．此外，还研制了一个5×5的传感器阵列并用于柔性透明的可穿戴式触摸键盘系统．研究结果表明所研制的压力传感器在未来的电子皮肤领域将具有良好的应用前景．

