ARTICLES SCIENCE CHINA Materials

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Published online 31 August 2023 | https://doi.org/10.1007/s40843-023-2534-1
Sci China Mater 2023, 66(10): 3922–3930

The piezoresistive pressure sensors based on ITO nanocrystalline-plant fiber composite

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ABSTRACT Wearable electronic devices have attracted extensive attention for their potential applications in robotic haptics, human-computer interaction, and human pulse wave measurement. A flexible pressure sensor, as a wearable electronic device, should be environmentally friendly and low-cost to manufacture. Cellulose paper-based pressure sensors have attracted much attention due to their excellent properties such as light weight, no or low toxicity, degradability, and flexibility. Here, we report a strategy for piezoresistive tactile sensors based on indium tin oxide (ITO) nanocrystal and plant fiber composite. The pressure sensor has a wide detection range (0–100 kPa), high sensitivity (464.88 kPa^{-1}), fast response time (6.93 ms) and recovery time (7.18 ms), and good loading and unloading stability. We also demonstrate that the as-prepared pressure sensors can be used for pulse testing, respiration monitoring, voice recognition, and various human motion detections. The results show that the pressure sensor based on the ITO nanocrystalline-plant fiber composite has the prospect of being applied to smart wearable electronic products.

Keywords: pressure sensors, piezoresistance, nanocrystalline, composite

INTRODUCTION

In recent years, flexible pressure sensors have attracted much attention for their great potential in robot haptics [1–3], health monitoring [4–7], wearable devices [8–11], and human-computer interaction [12–17]. In terms of the sensing mechanism, the main types are categorized into piezoresistive, capacitive, and piezoelectric pressure sensors. Among them, piezoresistive sensors have been widely studied due to their simple fabrication and sensing mechanism, i.e., by converting an external stimulus into a current signal and analyzing it. To develop piezoresistive tactile sensors with high sensitivity and good mechanical properties, sensing materials such as silver nanowires [18–20], carbon black [21–23], carbon nanotubes [22,24,25], and graphene [4,26–28] are usually used as dopants mixed in elastic host materials, or used as a conductor to contact flexible electrodes such as polyimide (PI) [29], polyethylene terephthalate (PET) [30,31], and polydimethylsiloxane (PDMS) [25,28,32]. However, these elastomeric or flexible electrode substrates are normally non-degradable and pose a threat to the environment.

To solve this problem, researchers have used biomass, such as cellulose [33,34], starch [35], and proteinas [36,37], as the host material or substrate to fabricate sensors. Among them, cellulose fibers are the most inexpensive as they are the most abundant of all plants [38]. The main product of cellulose fibers is paper, which is widely used in people's daily life and industry. The paper used in daily life has been widely recognized as an ideal platform for manufacturing low-cost, flexible, and environmentally friendly electronics. Paper-based flexible electronics [39–42] have the advantages of good flexibility, degradability, and recyclability as well as low cost. Paper-based pressure sensors have emerged as one of the most promising green electronic sensing devices. So far, many studies have been published on paper-based pressure sensors. For example, Tao *et al.* [43] prepared a graphene-paper pressure sensor using a graphene oxide suspension and an immersion coating process. Sakhuja *et al.* [12] designed a pressure sensor by designing a new type of multilayered paper structure through alternate stacking of plain and corrugated cellulose paper coated with the sensing material SnS. Yang *et al.* [44] developed a wearable strain pressure sensor using polymethyl methacrylate (PMMA) microspheres as a template to prepare porous graphene paper (PGP) as a sensing material. Qi *et al.* [45] prepared a high-performance and tear-resistant mulberry paper-based strain sensor by coating a graphene solution on mulberry paper through a simple Meyer bar coating process. Meanwhile, piezoresistive sensors functionalized by indium tin oxide (ITO) nanocrystals [46] have been less explored. The use of ITO nanocrystals for piezoresistive sensors or strain sensors is of great advantage due to the various advantages over the conventional materials mentioned above. The extremely high chemical environmental stability makes them promising for durable and sustainable applications, and thus, they can be ideal materials for a new generation of flexible wearable devices and electronics.

In this paper, we propose a method for fabricating a high-sensitivity pressure sensor using ITO nanocrystals as the conductive filler and plant fibers as the elastic matrix. The ITO nanocrystalline-plant fiber composite film is simply prepared by physical stirring and heating. The obtained pressure sensor has the advantages of a wide detection range (0–100 kPa), high sensitivity (464.88 kPa^{-1}), fast response (6.93 ms), fast recovery (7.18 ms), and good loading/unloading stability. Finally, the sensor is used to detect various physiological signals generated by the human body, such as pulse (or heartbeat), respiration, finger flexion, and facial muscle movement, fully demonstrating its application potential in medical health.

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EXPERIMENTAL SECTION

Materials

The materials we used included ITO nanocrystals (Xiamen Zhongli Tech. Co.), qualitative filter paper (Shanghai Titan Tech. Co.), interdigital electrodes etched on a conductive film of 100-nm-thick ITO-coated PET (Shenzhen South China Xiangcheng Tech. Co), and PI tape (Shanghai Lingguan New Material Tech. Co.).

Preparation of ITO nanocrystalline-plant fiber composite pressure sensors

Firstly, 0.02 g of qualitative filter paper scraps were added to 3 mL of deionized water and stirred for 4 h until the paper scraps became pulpy. Then, 0.2 g of ITO nanocrystals were added to the solution followed by stirring for 1 h. Using a pipette gun, 1 mL of the mixed solution was poured into a mold and dried into a film in an oven at 100°C (as shown in Fig. 1a), and then the film was cut to fit the active area of the ITO-PET interdigitated electrodes. Finally, the pressure sensor was encapsulated with PI tape (as shown in Fig. 1b).

Characterization

The morphology of the ITO nanocrystalline-plant fiber composites was characterized with scanning electron microscopy (SEM). Optical photographs of the filter paper and ITO nanocrystalline-plant fiber composites were taken using a digital camera. A tensile force tester (AIPU Digital Force Gauge HF-1) was used to apply different loads to the sensors. Real-time signals were obtained through a digital source meter (Keithley 2400). A piezoelectric ceramic actuator (PSt 150/5/7 VS10) was used to test the cyclic stability of the pressure sensor.

RESULTS AND DISCUSSION

Characterization of ITO nanocrystalline-plant fiber composite

As shown in Fig. 2a (×50) and b (×3000), the surface of the filter paper shows a randomly interconnected porous structure. The diameter of the fibers is much larger than the size of the ITO nanocrystals, thus providing abundant adhesion sites for the conductive ITO particles. The SEM images with different magnifications are shown in Fig. 2c–f, which further demonstrate the uniform distribution of ITO nanocrystals on the fiber surface with the stable interaction between the plant fibers and the ITO nanocrystals. The images with low magnifications, such as Fig. 2c (×100) and d (×2000), show that the ITO nanocrystals attached to the plant fibers partially fill the original holes in the fibers. The images with high magnifications, such as Fig. 2e (×30,000) and f (×50,000), show that the ITO nanocrystals can be stably and uniformly attached to the fibers. Therefore, there are at least two factors for the improvement of sensor sensitivity: One is the interior porous structure, which makes it very sensitive to external pressure, i.e., a small pressure can cause a large deformation; the other is that the well-conductive ITO nanocrystals have uniformly and firmly adhered to the surface of the elastic plant fiber, which not only provides good electrical conductivity but also provides a very loose connection between the conductive particles that are very sensitive to small pressure.

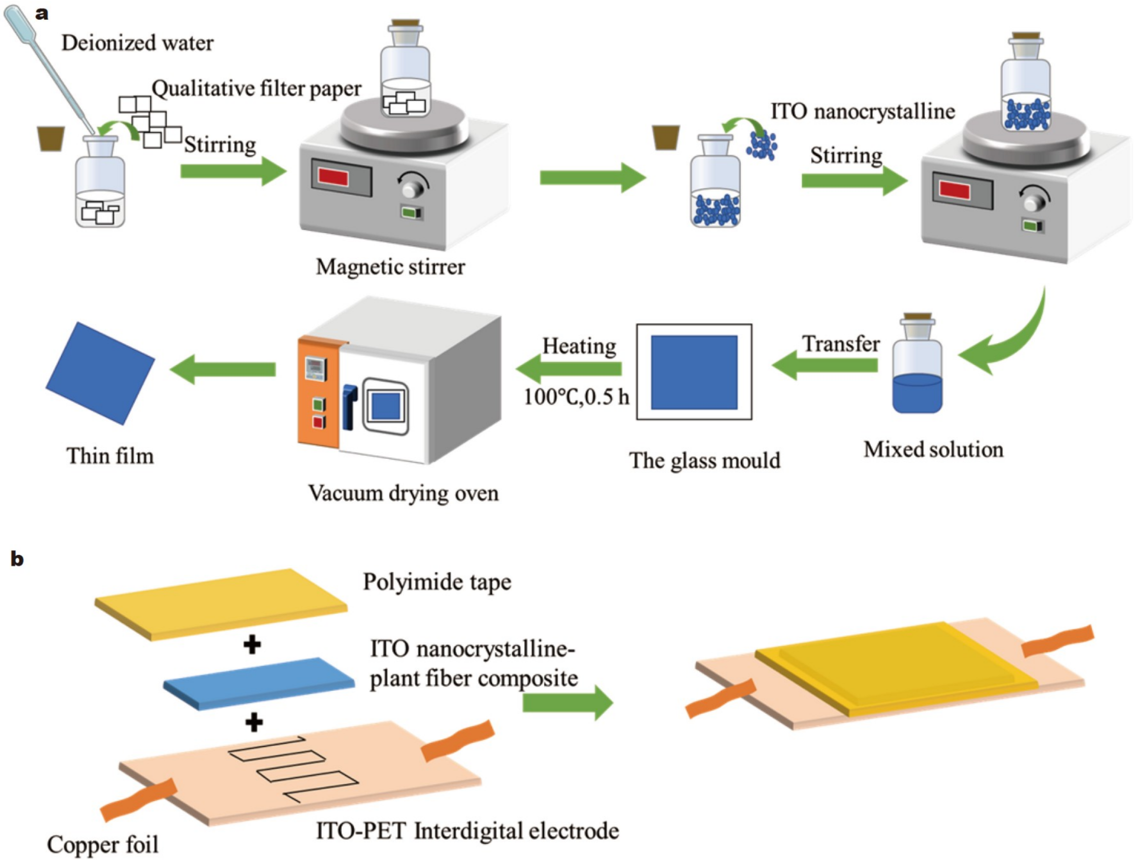

Figure 1 Schematic diagram of the preparation processes of ITO nanocrystalline-plant fiber composites (a) and the pressure sensor (b).

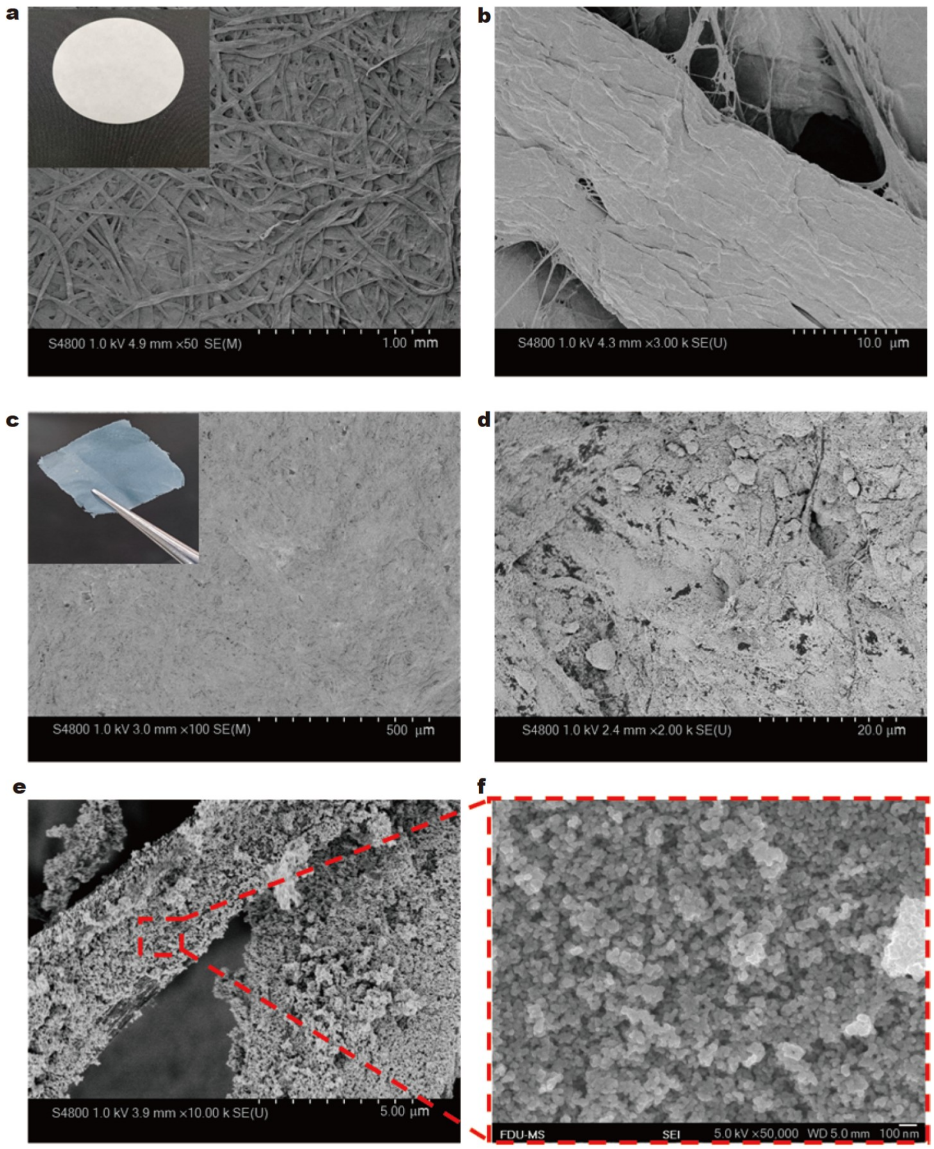

Figure 2 (a) Morphologies of qualitative filter paper (×50) and (b) ×3000. (c) Morphologies of the ITO nanocrystalline-plant fiber composite (×100), (d) ×2000, (e) ×10,000 and (f) ×50,000. The insets are the optical photographs.

Sensing performance of ITO nanocrystalline-plant fiber composite pressure sensors

The experimental setup for testing the pressure sensing performance of the ITO nanocrystalline-plant fiber composite sensor consists of the following four components: a sensor, a force gauge, a digital voltage source meter, and a computer for data collection, as shown in Fig. 3a. The sensing mechanism of the sensor is explained as follows. The plant fibers are hydrophilic and also have good shape controllability as well as flexibility of their own, which can be used as a supporting frame structure of the conductive material. After mixing ITO nanocrystals and plant fibers into the water, drying the film of conductive pulp, and forming a pressure-sensing conductive paper sandwiched in two electrodes, the sensor is completed. The ITO nanocrystals can be evenly attached to the surface of plant fibers. The ITO nanocrystals provide conductive sites, and the insulating plant fiber framework provides the elastic connection basis for the conductive sites. Thus the two constitute the pressure-sensitive sensing material. When a small compressing force is applied, the distance between the plant fibers, and the distance between ITO nanocrystals began to decrease, which first lead to an enhanced conductive path that allows sensitive detection of smaller pressures. Then, as the compression force increases, the distance between adjacent plant fibers gradually becomes smaller and the conductivity further increases with the expansion of the contact area between plant fibers, and between ITO nanocrystals, which corresponds to the pressure detection in a larger pressure range. The performance of this piezoresistive sensor can be optimized by adjusting the mass ratio between the plant fibers and the ITO nanocrystals, such as 1:3, 1:4, 1:6, and 1:10, to obtain the best pressure-sensing performance. In addition to flexibility, pressure sensors also require high sensitivity, optimal pressure response range, performance stability, reliability, etc. Excellent sensitivity (piezoresistive coefficient) means that a small pressure will result in a big current change in the sensor as the voltage keeps constant. Therefore, sensitivity was adopted as the most important

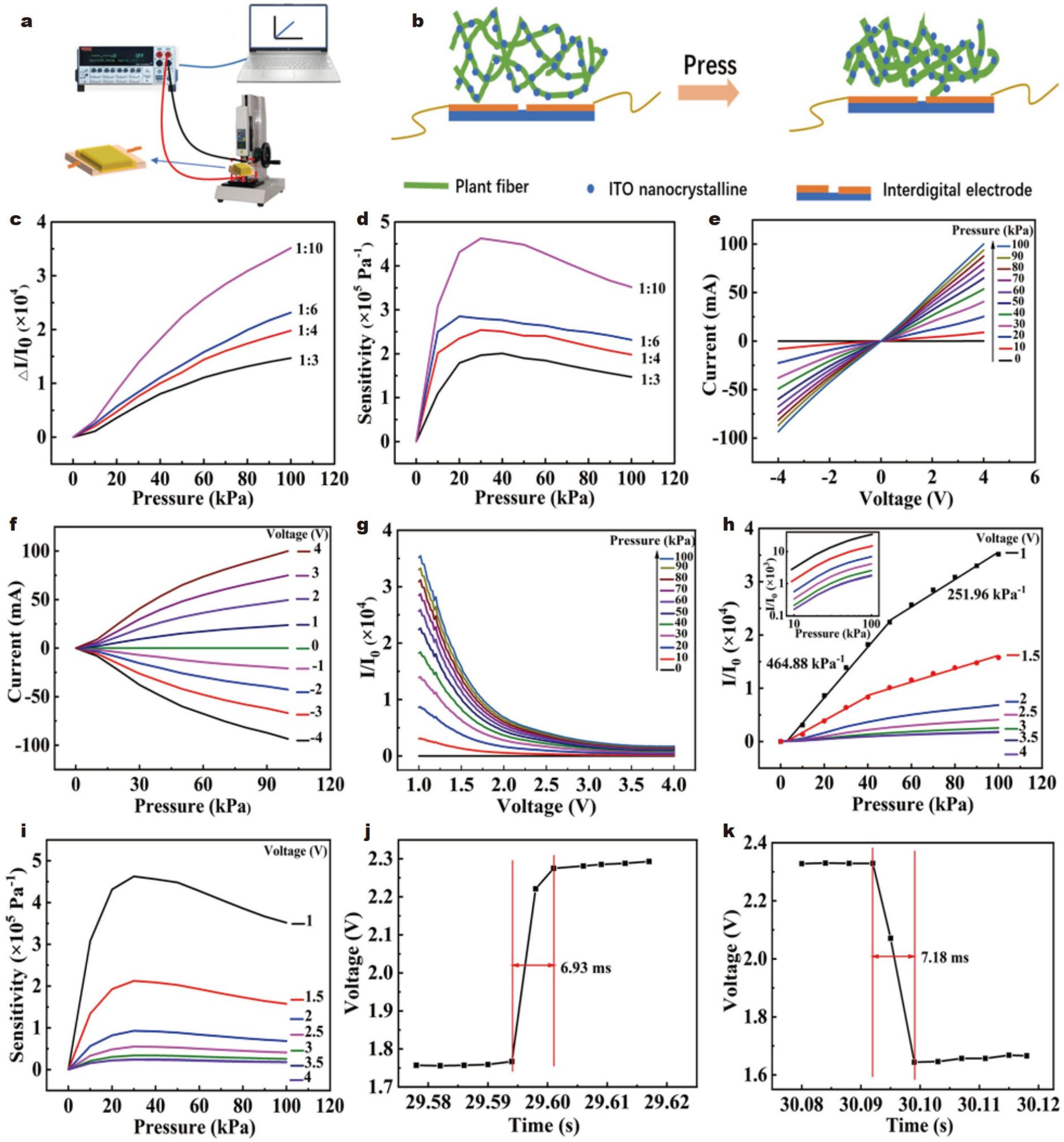

Figure 3 (a) Experimental setup; (b) schematic diagram of the sensing mechanism; (c) effect of the ratio of ITO nanocrystals on sensor performance; (d) pressure-dependent current amplification in devices with different ratios of plant fibers to ITO nanocrystals; (e) *I-V* curves at different pressures; (f) effect of different voltages on current with applied pressure; (g) *I-P* curves at different voltages; (h) current magnification varying with voltage at different pressures with the inset showing the logarithmic relationship for the ordinate; (i) current magnification varying with pressure at different voltages; (j) response time; (k) recovery time.

parameter to optimize the best mass ratio of ITO nanocrystals to plant fibers in the ITO nanocrystalline-plant fiber composite pressure sensor. As shown in Fig. 3c, d, the ITO nanocrystalline-plant fiber composite pressure sensor with a mass ratio of 1:10 exhibits the highest sensitivity to pressure. We then tested the *I-V* curves at different pressures. As shown in Fig. 3e, the current was varied at intervals of 0.01 V over the voltage range of −4 to 4 V, and it can be seen that the *I-V* curve is linear, indicating that the conduction of the sensor conforms to ohmic characteristics, and its slope is the reciprocal of the device resistance. Fig. 3f shows the current variation curves of the sensor with pressure at different voltages. When the current of its negative voltage part is taken as the absolute value, the current-pressure (*I-P*) curves of the sensors under positive and negative voltages of the same magnitude are almost identical, indicating that the sensors are not affected by voltage directionality. So both positive and negative voltages can be used. The current amplification curves of the sensor with voltage at different pressures are shown in Fig. 3g. At an applied pressure of 100 kPa and a voltage of 1 V, the magnification is 3.6×10^4, which indicates that the current changes significantly with the applied pressure. The ratio of the current amplification factor to the pressure change factor in the device, that is, the piezoresistive coefficient, is one of the most important parameters for evaluating sensor performance. As shown in Fig. 3h, at different voltages, the amplification factor of the current increases with the applied pressure. Take the logarithmic relationship for the ordinate of Fig. 3h, as shown in the inset. As the pressure increases, the tendency of the current increase tends to saturate. Fig. 3i shows the device piezoresistive coefficient as a function of pressure from 0 to 100 kPa. The piezoresistive coefficient first increases and then decreases with the increase of pressure, and decreases with the increase of voltage, so the sensor can be applied to the perception of small pressure under low voltage. The piezoresistive coefficient (S) is calculated as

$$S = (\Delta I/I_0)/\Delta P, \quad (1)$$

where ΔI is the relative change in current, I_0 is the no-load current, and ΔP is the change in applied pressure. For the ITO nanocrystalline-plant fiber composite pressure sensor, the sensitivity at low voltage is higher than that at high voltage, as shown in Fig. 3h. In the low-pressure range (0–50 kPa), the sensitivity is about 464.88 kPa^{-1}, while in the high-pressure range (50–100 kPa), the sensitivity drops from 464.88 to 251.96 kPa^{-1}. As the loading pressure increases, the sensitivity of the sensor gradually decreases. Under the initial loading pressure, the plant fibers with ITO nanocrystals attached to the surface contact each other, resulting in a larger change in the ITO-connected area and thus exhibiting higher sensitivity on resistance. When the pressure is continuously increased, the ITO-connected plant fiber causes a smaller change in the contact area, and thus causes a smaller change in current under high pressure, showing a lower sensitivity. The pressure sensor can work in a wide pressure range (0–100 kPa) and maintain good sensitivity. Response and recovery time are also extremely important parameters for a sensor. A force of 20 kPa was applied to the sensor in the form of a square wave, then a response time of approximately 6.93 ms and a recovery time of approximately 7.18 ms were obtained, as shown in Fig. 3j, k. This means that the sensor can respond to pressure quickly, and also return to the initial shape quickly.

Stability of ITO nanocrystalline-plant fiber composite pressure sensors

To test the stability and reversibility of the pressure sensor, we applied the pressure from 0 to 100 kPa with a 10-kPa step, gradually increasing at first and then decreasing under the square wave voltage, as shown in Fig. 4a. It can be seen that the current of the pressure sensor can respond to the electric field very fast, and also responds quickly to the alternating increase and decrease in pressure, indicating that the prepared pressure sensor has good stability and repeatability. The current varying with time was measured under different pressures (Fig. 4b) for studying the stability and reliability of the piezoresistive sensor. The current almost keeps stable under a fixed pressure, indicating that the pressure sensor has a stable resistance at different pressures. In addition, the response characteristics of the sensor were investigated by varying the loading frequency. In Fig. 4c, the piezoresistive sensor shows the same current response at four loading frequencies (0.2, 0.3, 0.5, and 0.7 Hz). The maximum values of their currents are essentially the same, indicating that the pressure sensor has stable performances and good dynamic responses at the four measured frequencies. As shown in Fig. 4d, the measured current varied with elapsing time when the sensor was applied a 3-V bias and was pressed at a depth varying as a time-dependent function of a sine wave, a triangle wave, a square wave, and a sawtooth wave. The indented depth varying with time as the function of the sine wave, triangle wave, square wave, and sawtooth wave can be distinguished roughly from the measured time-dependent current. In addition, to further explore the repeatability and durability of the sensor, 700 loading and unloading cycles were tested at an applied pressure of 10 kPa and a frequency of 1 Hz. As shown in Fig. 4e, the response current remains essential constant. To see more clearly the curve of its internal current during the loading-unloading processes, the curve at one stage was scaled, as shown in the inset.

Furthermore, we explored the effects of paper types. Due to the different raw materials and processes used in the paper manufacturing process, the internal microstructure of the plant fibers is different, which affects the performance of the manufactured sensors. We selected four kinds of paper commonly used in daily life: filter paper, printing paper, dust-free paper, and roll toilet paper. They were used to fabricate sensors, and the performances of the four devices were compared. From Fig. 5a, b, it can be seen that the sensing material prepared with filter paper fibers has the best performance. The reason may be that the fibers of the filter paper are slender, twisted from fibers ranging from tens to hundreds of nanometers, and the structure is full of pores. Printing paper, laboratory dust-free paper, and roll toilet paper are mostly composed of coarse fibers of 10–30 μm, and the space between fibers is larger.

Applications of ITO nanocrystalline-plant fiber composite pressure sensors

As the ITO nanocrystalline-plant fiber composite pressure sensor features fast response recovery time, excellent stability, and sufficient sensitivity, we investigated its performance in real-time human monitoring applications. By using medical tape, the ITO nanocrystalline-plant fiber composite pressure sensors can be tightly attached to various parts of the body. Firstly, the pressure sensor was used to detect the slight movement of the body. As shown in Fig. 6a, b, the sensor can sense the corresponding pulse wave signal due to the change over time in blood pressure in the radial and carotid arteries. The pulse of the radial artery of the wrist, as shown in Fig. 6a, has a waveform that is consistent with the pulse wave of a reference artery obtained according to medical standards. In addition, the pressure sensor can detect the intensity and frequency of our breathing. The device can be used as the mask worn on the face of the tester to detect the weak pressure caused by the exhaled airflow during breathing. As can be seen from the current-time curve shown in Fig. 6c, the respiratory rate of the tester was recorded at 18 min^{-1}, which is in line with the actual level. The ITO nanocrystalline-plant fiber composite pressure sensor can detect the vibration signal of vocal cords for speech recognition, as shown in Fig. 6d. The current-time curves show significant differences and good repeatability when pronouncing different words: "tomorrow" and "hello". The pressure sensor is connected to the index finger joint, which allows for the measurement of finger flexion and recovery. As can be seen in Fig. 6e, the current shows a fast and stable response to the finger at different angles. As the finger bending angle gradually increases, the magnitude of the average current change generated by the pressure sensor increases accordingly. When the sensor was attached to the

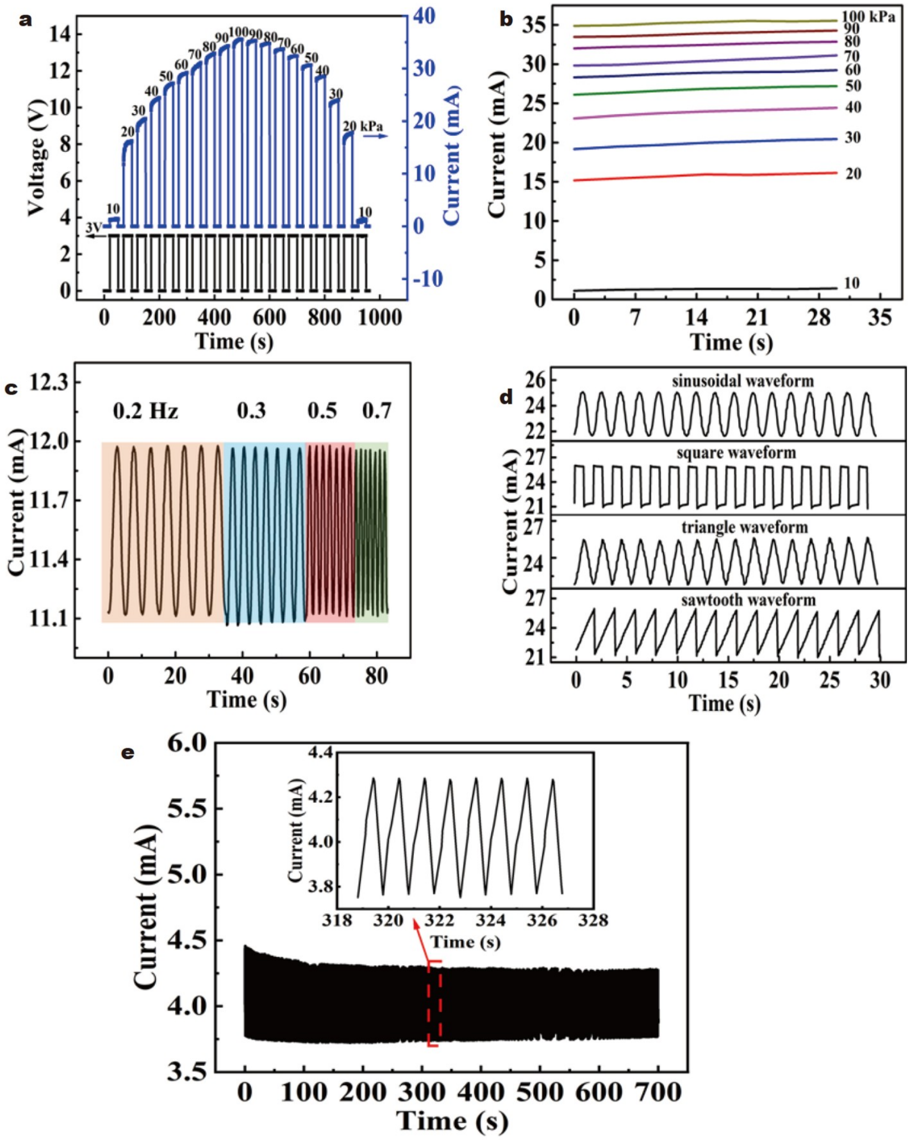

Figure 4 (a) Current of the sensor varies with time under different pressures and square wave voltages with 0 V (low level) and 3 V (high level). (b) The current varies with the elapsing time under different pressures and a stable voltage of 3 V. (c) Current response from different loading frequencies. (d) Current responses from the indented depth which is varying with the time-dependent function of a sine wave, a triangle wave, a square wave, and a sawtooth wave. (e) Sensor current varies with time under a stable voltage of 3 V, while the sensor is pressed and the indented depth follows a time-dependent triangular wave function.

tester's right arm, as shown in Fig. 6f, the current changed significantly as the arm changed from a relaxed state to a clenched fist state, indicating that it accurately identifies muscle movement. The sensor can also potentially be applied to detect large deformations caused by human movement, including wrist bending, knee bending, and elbow bending. The sensor showed stability under various human motions, proving that the sensor has broad prospects in the field of smart wearable devices.

In order to obtain a tactile sensor with a wide detection range, high sensitivity, fast response time/recovery time, and good loading and unloading stability, many factors need to be considered. For example, a wide detection range requires sufficient materials with a wide range of deformation to participate in sensing and the working pressure range is large enough; the high sensitivity of the device requires a relatively small working pressure, that is, the material participating in the deformation responds to small pressure and has good elasticity, and at the same time the signal change is large enough. In terms of the piezoresistive sensing mechanism of the device, it is the contact area between the conductive particles needs to increase with the pressure increasing and the distance decreasing. The fast response time/recovery time requires that the material has little creep, the shape rebounds quickly, and the material plasticity is very low. The electrode of the device needs to be strong and firm enough to ensure stable loading and unloading, so the sensor structure can adopt channel-structure instead of sandwich-structure. The deformation process of the piezoresistive sensing material with the pressure is stable and fully recoverable, without creep and plasticity. We have tried many composites and find that the comprehensive performance of the composite material

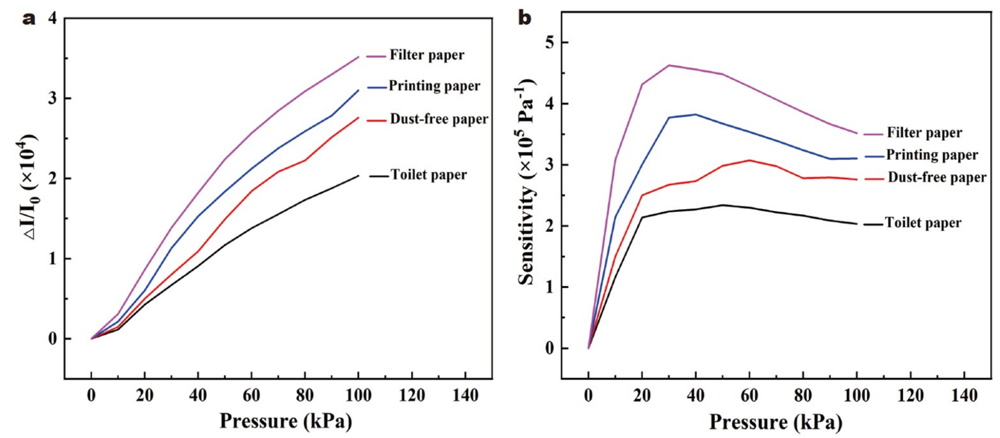

Figure 5 Pressure-dependent current amplification (a) and sensitivity (b) of sensors based on four kinds of paper: filter paper, printing paper, dust-free paper, and toilet paper.

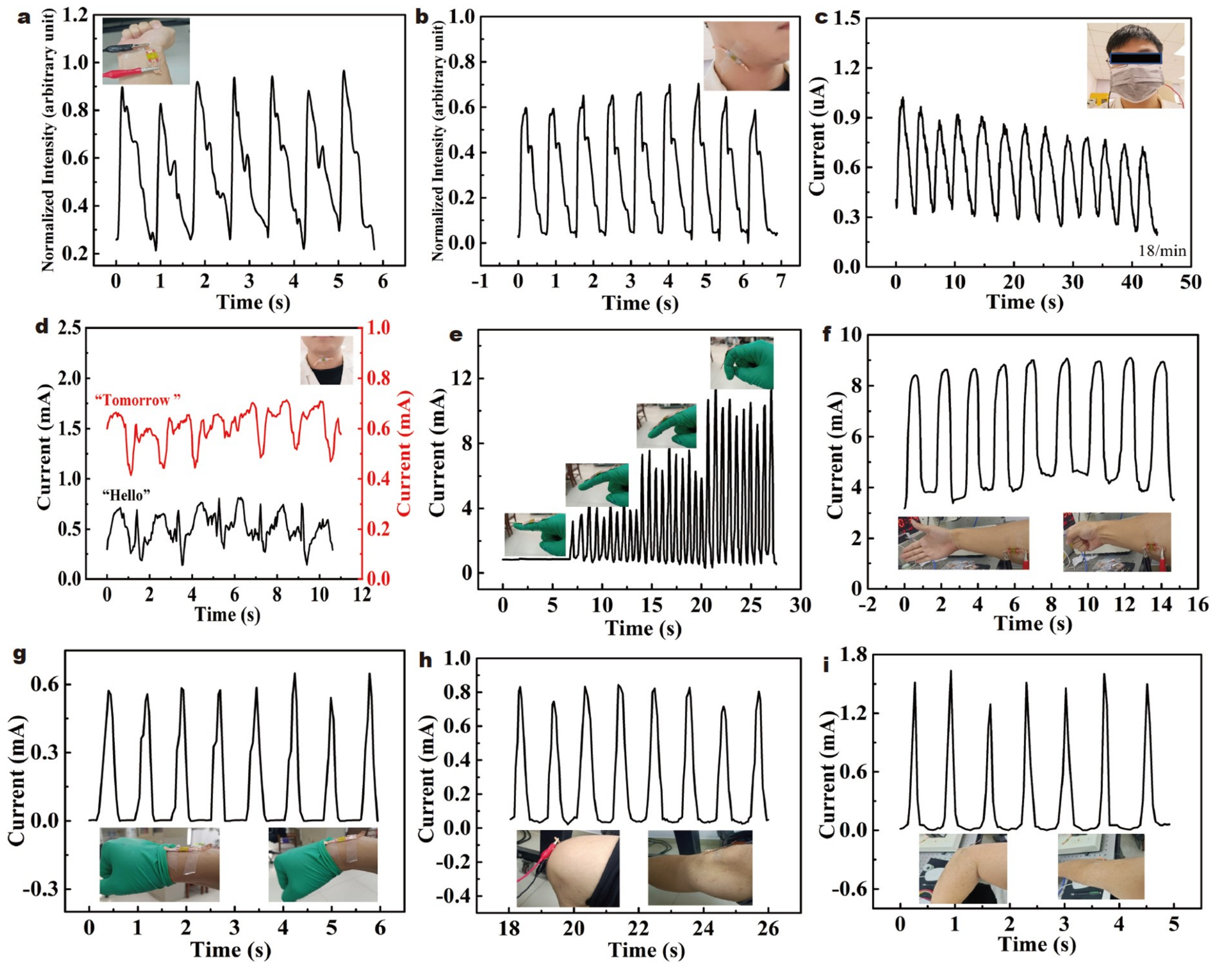

Figure 6 Monitoring signals based on ITO nanocrystalline-plant fiber composite pressure sensors for various human movements: (a) wrist pulse; (b) neck pulse; (c) breathing monitoring; (d) voice recognition; (e) finger bending; (f) arm bending; (g) wrist bending; (h) knee bending; (i) elbow bending.

in this study is the best.

Comparison with similar sensors

The piezoresistive pressure sensors based on ITO nanocrystalline-plant fiber composite demonstrate superior performance in the sensitivity and sensing ranges. As shown in Fig. 7, we compared our pressure sensor with other piezoresistive sensors reported in literature [8,12,40,47–53]. It can be seen that there is a compromise between the general sensor sensitivity and sensing range. Piezoresistive sensors with ultrahigh sensitivity tend to have a narrow sensing range, but in order to achieve a wide sensing range, sensitivity is generally sacrificed. In contrast, the

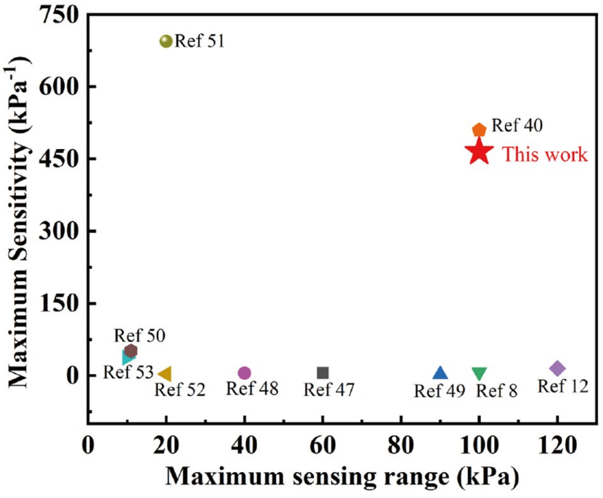

Figure 7 Comparison of sensor sensitivity *versus* pressure range for flexible piezoresistive sensors.

piezoresistive pressure sensors based on ITO nanocrystalline-plant fiber composite proposed in this work exhibit an excellent sensitivity of 464.88 kPa^{-1} in the region of 0–50 kPa and 251.96 kPa^{-1} in the range of 50–100 kPa.

CONCLUSIONS

In summary, we propose a simple and environmentally friendly method to prepare ITO nanocrystalline-plant fiber composite pressure sensors. The piezoresistive sensor is based on a composite of ITO nanocrystals and plant fibers, which is made by mixing ITO nanocrystalline and paper into water to form ITO-doped pulp, and then dried to form a pressure-sensing conductive paper. This sensor can be used for large-scale production. The prepared ITO nanocrystalline-plant fiber pressure sensors show high sensitivity (up to 464.88 kPa^{-1}), wide operating range (0–100 kPa), fast response (6.93 ms), rapid recovery (7.18 ms), and good repeatability. The sensor has been applied to detect physiological activity (pulse test) and many different human movements, such as finger bending, arm bending/extending, and distinguishing different words from different pronunciations. This sensor demonstrates the application prospect in the field of smart wearable devices, which can be manufactured simply, mass-produced at low-cost, and used in health monitoring.

Received 28 April 2023; accepted 27 June 2023;
published online 31 August 2023

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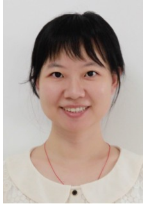
Acknowledgements This work was financially supported by the National Natural Science Foundation of China (11874267 and 51373036) and the National Science Foundation for Young Scientists of China (61704107).

Author contributions Xia Y designed and participated in the whole project; Huang P fabricated and measured the samples; Lin X, Wu L, and Li K characterized the devices; Gao C fabricated the devices; Huang P and Zhong G wrote and revised the manuscript. All authors contributed to the general discussion.

Conflict of interest The authors declare that they have no conflict of interest.



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ITO纳米晶-植物纤维复合材料的电阻式压力传感器

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摘要 可穿戴电子设备因其在机器人触觉、人机交互和脉搏测试等领域具有潜在应用而引起广泛关注. 作为一种可穿戴电子设备, 柔性压力传感器应该是环保的, 而且制造成本低. 基于纤维素纸的压力传感器由于其优良的特性, 如轻质、无毒或低毒、可降解性和柔韧性, 而引起了人们的关注. 在此, 我们报道了一种基于铟锡氧化物(ITO)纳米晶体和植物纤维复合材料的电阻式触觉传感器. 该压力传感器具有宽的检测范围(0–100 kPa)、高灵敏度(464.88 kPa^{-1})、快速的响应时间(6.93 ms)和恢复时间(7.18 ms)以及良好的加载/卸载稳定性. 我们还展示了该压力传感器用于脉搏测试、呼吸监测、语音识别等各种人体运动检测. 结果表明, ITO纳米晶-植物纤维复合材料压力传感器在智能可穿戴电子产品中显示出巨大应用潜力.