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Flexible and High Performance Piezoresistive Pressure Sensors Based on Hierarchical Flower-Shaped SnSe₂ Nanoplates

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Abstract

Pressure sensors with high sensitivity, broad dynamic response range and short response time are highly desirable to enable applications into robotics and artificial intelligence. Here, a high-performance piezoresistive pressure sensor based on conical frustum-like structured polydimethylsiloxane (PDMS) and naturally formed flower-shaped SnSe₂ nanoplates is demonstrated. The structured PDMS/Au and SnSe₂ nanoplates/Au interdigital electrodes are exploited as the top and down electrodes, respectively. The proposed configuration shows a significantly enhanced sensitivity (433.22 kPa⁻¹), when compared with conventional configuration (flat Au film as the bottom interdigital electrodes). The resulting pressure sensor (PDMS/Au/Au/SnSe₂) presents broad dynamic response range (0~38.4 kPa), low detection limit (~0.82 Pa), short response time (~90 μs), and long-term cycle stability (>1000 cycles). Furthermore, large-area sensor arrays are successfully fabricated and can be used to detect both the intensity and distribution of the loaded pressure. Thanks to these merits, we believe that our high-performance pressure sensor is an ideal candidate for practical applications in robotics, smart devices and artificial intelligence.

Introduction:

Pressure sensor is an inevitable electronic device which enables the efficient conversion from the mechanical stimuli to the electronic signals, with applications in soft robotics, smart devices and artificial intelligence¹⁻⁴. In recent years, pressure sensors with various sensing mechanisms have been demonstrated, including capacitive, piezoelectric and piezoresistive sensing⁴⁻¹⁴. Among these technologies, the piezo-resistivity sensing is more promising and widely used due to the simplicity of design, low manufacturing cost, good signal repeatability and high sensitivity.

By far, a wide variety of materials (such as two-dimensional transition metal dichalcogenide, graphene, polymers) and structures (nanosheets and nanowires) have been demonstrated for high-sensitivity piezoresistive pressure sensors¹⁵⁻²⁴. For a typical piezoresistive pressure sensor, the sensitivity is determined by current or contact resistance change with pressure, as well as being proportional to the variation of the sensor contact area. An effective strategy to improve the pressure sensors' sensitivity is to introduce fine microstructure features, which result in a larger variation of the contact area, compared to the pressure sensors with flat structures. Creating surface textures on polydimethylsiloxane (PDMS) is a common way to form highly sensitive pressure sensors. For example, it is found that PDMS structures with micropyramids, micro-hemispheres, micro-semicylinders and microhump patterns (*via* sandpaper mold) can improve the sensors' sensitivity²⁵⁻²⁷. In these pressure sensors, the resistance change is caused by the deformations of the microstructures under the external pressure. However, most of the previous works only focus on the design of the top PDMS microstructures, and challenges remain on the development of pressure sensors that can combine the merits of high sensitivity, fast response and low detection limit. In principle, the total contact area of a sensor is the product of the touch points and the contact area of individual microstructures. Therefore, it is expected that introducing contact touchpoint control in both the top and down sensor structures will increase the variation of the contact area, which in turn enhances the performance of the pressure sensors.

In nature, there are a wide range of well-adapted structures developed by the process of natural selection, which have inspired us for the design of pressure sensor devices²⁸⁻³⁰. For example, Cao et al reported a piezoresistive pressure sensor inspired by the epidermal-dermal and the outer microstructures of the human fingerprint and demonstrated a high sensitivity of -3.26 kPa^{-1} in the pressure range of 0-300 Pa²⁸. Xia

et al proposed a high performance pressure sensor based on fingerprint-like patterned 3D graphene film *via* chemical vapor deposition. The sensor showed a high sensitivity of 110 kPa^{-1} for 0-200 Pa and fast response ($< 30 \text{ ms}$)³⁰.

In this work, we present a high performance piezoresistive pressure sensor that employs the naturally formed flower-shaped SnSe₂ nanoplates/Au as the bottom part and the conical frustum-like structured PDMS/Au as the top part. Compared to the devices with flat bottom Au electrodes, the proposed configuration shows a significantly enhanced sensitivity. The sensing devices offer superior sensitivity (433.22 kPa^{-1}), broad working pressure range (0~38.4 kPa), fast response ($\sim 90 \mu\text{s}$) and very low detection limit (0.82 Pa). In addition, large-area sensor arrays are successfully fabricated and can be used to detect both the intensity and distribution of the loaded pressure.

Results and discussion

Figure 1a schematically illustrates the key process steps for the fabrication of the PDMS/Au/Au/SnSe₂ pressure sensor. The top microstructures are transferred onto the flexible PDMS mold by simple lithography and casting process, followed by the deposition of Au film (more details are available in the Experimental Section). The SnSe₂/Au interdigital electrodes are prepared *via* co-evaporation of SnSe₂ nanoplates³¹, the deposition of Au film and laser scribing process (more details are available in the Experimental Section). Then, the device is formed by assembling PDMS/Au and SnSe₂/Au interdigital electrode. Figure 1b-c are the scanning electron microscopy (SEM) characterization of the obtained SnSe₂ film, showing the naturally formed flower-shaped SnSe₂ nanoflakes with layered structures. This morphology is formed through a homogenous nucleation process and the subsequent formation of flower-shaped SnSe₂ nanoflakes which strongly anchor on the glass surface (Figure S1). Figure 1d depicts the corresponding atomic force microscope (AFM) image. The peak to peak roughness is $\sim 350 \text{ nm}$. Figure 1e-g and figure S2 are the SEM and AFM images of the resulting patterned PDMS mold, showing the conical frustum-like microstructures. Figure 1h-j depicts the optical microscopy image of the SnSe₂/Au interdigital electrodes *via* laser scribing process. The width of a single interdigital is $\sim 120 \mu\text{m}$, the interval between two neighboring interdigital electrodes is $\sim 350 \mu\text{m}$.

The crystallinity and composition of SnSe₂ nanoplate are also investigated by XRD, Raman spectroscopy and EDS, as shown in Figure 2. Figure 2a shows the XRD pattern of SnSe₂. The curve matches well with the hexagonal SnSe₂, which indicates a layered structure in which Sn layers are sandwiched between two Se layers facing each other (shown in the inset of Figure 2a)³². Figure 2b is the Raman spectra of the SnSe₂ nanoplate on glass substrate, showing two notable Raman peaks at 114.5 and 184.7 cm^{-1} . The former peak is the E_g mode and the latter one is the A_{1g} mode, which are due to the in-plane and out-of-plane vibrations (shown in the inset of Figure 2b), respectively³³. The elemental ratio Sn: Se in the nanoplate is close to 1:2, according to the EDS analysis (Figure 2c-d). Under the external pressure, the top hierarchical, conical frustum-like

structured PDMS and the bottom nature-patterned SnSe₂ microstructures with rich sharp peaks provide lots of effective contact sites and sufficient roughness to “feel” the variation of contact resistance, which in principle can improve the performance of the pressure sensing devices.

To demonstrate the improved performance of the proposed structure, two types of pressure sensors including PDMS/Au/Au structure (the peak-to-peak roughness of the fabricated smooth Au interdigital electrode is ~4.5 nm, as shown in Figure S3) and PDMS/Au/Au/SnSe₂ structure are fabricated. Figure 3a plots the representative response curves of the pressure sensors with flat Au sample and microstructured SnSe₂/Au sample. The sensitivity of a pressure sensor can be defined as $S = ((\Delta I/I_0)/\Delta P)$, where ΔI is the current change and $\Delta I = I_P - I_0$, where I_P is the measured current under loading and I_0 is the current of the sensor under the minimum measurable load. The sensor sensitivity S can be obtained by the linear slope of the plot before the current saturation. Similar to most of the reported pressure sensors, the plot for sensitivity can be divided into two-regions (low- and high- pressure ranges). The measured sensitivity is 11.52 (0.07) kPa⁻¹ and 433.22 (2.91) kPa⁻¹ in the low (high)-pressure ranges for the sensors with PDMS/Au/Au and PDMS/Au/Au/SnSe₂ configurations, respectively. Compared with PDMS/Au/Au configuration, the PDMS/Au/Au/SnSe₂ configuration offers the higher sensitivity and broader working pressure range. This dramatic increase in the sensitivity of the PDMS/Au/Au/SnSe₂ configuration over the PDMS/Au/Au configuration can be attributed to the introduction of height variations for both top PDMS microstructures and bottom SnSe₂/Au interdigital electrodes. As shown in Figure 3b, in the PDMS/Au/Au configuration, the current change is due to the deformation of the PDMS microstructures during the pressure loading process, which leads to the increase of the contact area of each individual microstructure. In comparison, in the PDMS/Au/Au/SnSe₂ configuration, the initial contact is formed by the highest microstructures on the top PDMS and the tips of the SnSe₂ nanoplates on the bottom interdigital electrodes, thus resulting relatively high initial resistance. Under the external pressure, the highest microstructures begin to deform, while the other lower PDMS microstructures start to touch the SnSe₂ nanoplates. Thus, the contact touchpoints significantly increase and in turn decreases the contact resistance of the sensor, which finally contributes to its ultrasensitive features. Furthermore, in our PDMS/Au/Au/SnSe₂ configuration, even after all PDMS microstructures fully touch the bottom SnSe₂/Au electrodes, the sensor is still functional with the resistance change due to the microstructure deformation under pressure.

Stability under cyclic deformations is a basic requirement for the pressure sensor. Therefore, we investigate the durability of the PDMS/Au/SnSe₂ pressure sensor. As shown in Figure 3c, the relative current changes remain about the same value. Inset of Figure 3c shows that there is no obvious current degeneration during the whole cyclic process, which is greatly important for the long-term usage. Fig. 3d exhibits the consecutive measurements of the PDMS/Au/Au/SnSe₂ pressure sensor under a wide range of different on/off pressure: 40 Pa, 160 Pa, 4000 Pa and 12000 Pa. The current increases quickly after the pressure is applied and recovers to the original level when the pressure is removed. Also, the relative changes in resistance precisely follow the

variation of the external pressures from 40 Pa to 12000 Pa, indicating the great stability and reproducibility of the PDMS/Au/Au/SnSe₂ pressure sensor upon various pressures regardless of pressure regions. Figure 3e is the current-voltage (I-V) curves of the PDMS/Au/Au/SnSe₂ pressure sensor under different pressures from 30 Pa to 1200 Pa. All curves show good linearity in the voltage form -5 V to 5 V, implying its good ohmic behavior. Therefore, the sensitivity of the PDMS/Au/Au/SnSe₂ pressure sensor is independent of the working voltage. In addition, as the applied external pressure increases, the slope of the I-V increases, showing that the resistance decrease as the external pressure increases. Figure 3f depicts the instant response of the PDMS/Au/Au/SnSe₂ pressure sensor. Under the pressure of 20 Pa, the response and recovery time the PDMS/Au/Au/SnSe₂ pressure sensor are ~70 μ s and ~90 μ s, respectively. The fast response speed is superior to many reported pressure sensors, as shown in Table S1.

Our PDMS/Au/Au/SnSe₂ pressure sensor is ultrasensitive for the detection of weak pressure changes. Figure 3g-h shows that the PDMS/Au/Au/SnSe₂ pressure sensor can detect a soybean with the pressure of only 0.82 Pa, indicating a detection limit lower than many previous reported pressure sensors (See Table S1 in the supporting information). This is due to the ultrahigh sensitivity property of the PDMS/Au/Au/SnSe₂ pressure sensor in the low-pressure region (increment of point contacts). Moreover, our PDMS/Au/Au/SnSe₂ pressure sensor possesses the ability to resolve the magnitude and spatial distribution of various complex objects with arbitrary shape. Figure 4a and 4d show the schematic illustration of the designed 3 * 3 and 4 * 4 pixel arrays of SnSe₂/Au interdigital electrodes. Figures 4b-c and Figures 4e-f show the demonstrations with a right-angle-type hexagon wrench and different complex objects (screw, nut, prill, et. al.), respectively, indicating that the sensor array can perceive the load shape, load pressure and differentiate the compressed local regions from other regions.

Conclusion

In conclusion, we have demonstrated a piezoresistive pressure sensor based on conical frustum-like structured PDMS and flower-shaped SnSe₂ nanoplates. The resulting pressure sensors exhibit high sensitivity 433.22 kPa⁻¹, broad dynamic response range (0~38.4 kPa), fast response ($\approx$ 90 μ s) and low detection limit (0.82 Pa). Furthermore, large-area integration of these PDMS/Au/Au/SnSe₂ pressure sensors can be easily fabricated for mapping and identifying the spatial pressure distribution. These remarkable features of our pressure sensors offer great potential for soft robotics, smart devices and artificial intelligence applications.

Experimental Section

Preparation of microstructured PDMS/ Au.

First, AZ4620 photoresist (AZ-4620) thin film was spin-coated on the glass substrate at the speed of 500 rpm and 3000 rpm for 10 s and 30 s, respectively. Then the sample was baked at 100 °C for 6 min, followed by an exposure for 80 s. After the exposure, the sample was baked at 120 °C for 3 min, followed by the development process for 3 min. Then the sample was baked at 100 °C for 5 min,

finally forming a photoresist thin film with certain holes on the glass substrate.

The PDMS liquid was prepared as follows: the silicone elastomer and curing agent with mass ratio of 10:1 were mixed and stirred for 20 min at room temperature. Then the mixed liquid was degassed in vacuum at room temperature for 30 min. The microstructured PDMS was prepared as follows: the mixed liquid was spin-coated on the photoresist mold at the speed of 400 rpm for 30s. Then the sample was baked at 80 °C for 2 h. The photoresist was then removed by acetone. After demolding, the patterned side of PDMS was further rinsed by acetone, followed by drying at room temperature for 30 min. In the end, Cr/Au with thickness of 1 nm/ 100 nm were deposited on the patterned side of as-prepared PDMS by thermal evaporation.

Preparation of flower-shaped SnSe₂ nanoplates/ Au interdigital electrode.

Flower-shaped SnSe₂ nanoplates was deposited employing co-evaporation process in a molecular beam epitaxy system. The background pressure of the evaporation system was 5×10^{-5} Pa. The heating temperature for Sn, Se and the glass substrate were 1100 °C, 235 °C and 250 °C, respectively. The deposition time was 20 min. After the synthesis of SnSe₂ nanoplates, Cr/Au with thickness of 1 nm/ 100 nm were deposited on the SnSe₂ nanoplates. The SnSe₂ nanoplates /Au interdigital electrode was then fabricated by laser scribing process.

Characterization and Performance Testing:

For SEM and EDS measurements, ZEISS SUPPA55 equipped with Oxford EDS detector was used. For Raman analysis, Witec CRM200 system equipped with a solid-state YAG laser (532 nm) was used. For XRD, Rigaku D/ Max-2500 diffractometer was used, the scanning rate is 5° min⁻¹. For AFM measurement, Agilent 5500 was used. For the pressure performance measurement, the testing system is as shown in Figure S4. The current-time-pressure characteristics were determined using Keithley 2400, the pressure meter (DS2-5N, Dongguan Zhiqu Precision Instruments Co.; Ltd.) and the oscilloscope (LUCK-3, digital storage oscilloscope, Chengdu Rongte instrument Co., Ltd)

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Figure 1 | PDMS/Au/Au/SnSe₂ pressure sensors. **a**, Schematic illustration of the fabrication process of the PDMS/Au/Au/SnSe₂ pressure sensors. **b-d**, Top-view SEM and AFM images of the flower-shaped SnSe₂ nanoplates. **e-g**, Cross-sectional and Top-view SEM images and height profile of the conical frustum-like PDMS microstructures. **h-j**, Optical and SEM images of the rough SnSe₂/Au interdigital electrodes.

Figure 2 | Spectroscopic characteristics of SnSe₂ nanoplates. **a**, X-ray diffraction pattern of SnSe₂ nanoplates on glass substrate. **b**, Representative Raman spectra of SnSe₂ nanoplates on glass substrate. **c-d**, SEM image of the obtained SnSe₂ nanoplates and the corresponding Sn and Se elemental mapping. The inset of **d** is the EDS spectrum of the SnSe₂ nanoplates. The elemental ratio Sn: Se is 32.18: 67.82, which is close to 1:2.

Figure 3 | Pressure-sensing capabilities of the PDMS/Au/Au/SnSe₂ pressure sensors. **a**, The comparison of pressure sensitivities of different sensor structures: PDMS/Au/Au (red), PDMS/Au/Au/SnSe₂ (blue). **b**, Working mechanism of the

PDMS/Au/Au and PDMS/Au/Au/SnSe₂ pressure sensors: schematic illustrations of conduction path models under different loading state. **c**, Real-time I-t curves of the PDMS/Au/Au/SnSe₂ pressure sensor for more than 1000 loading/unloading cycles with an applied pressure of 25 Pa. **d**, Time-resolved static current change response of the PDMS/Au/Au/SnSe₂ pressure sensor under repeated mechanical loads, with different pressures ranging from 40 Pa to 12 kPa. **e**, Current-voltage (*I-V*) curves of the PDMS/Au/Au/SnSe₂ pressure sensor with different applied pressures. **f**, Response and recovery time of the PDMS/Au/Au/SnSe₂ pressure sensor. **g-h**, Detection of weak pressure: Optical image and current curve of the proposed PDMS/Au/Au/SnSe₂ pressure sensor pressed by a soybean. Weak pressures can be detected by the PDMS/Au/Au/SnSe₂ pressure sensor, and the detection limit reaches 0.82 Pa.

Figure 4 | Pressure mapping of complex objects on PDMS/Au/Au/SnSe₂ pressure sensor arrays. **a-c,** Current mapping for the pressure of a heavy right-angle-type hexagon wrench by 3*3 PDMS/Au/Au/SnSe₂ pressure sensor arrays. **d-f,** Current mapping for the pressure of some complex objects (screw, nut, prill, et. al.) by 4*4 PDMS/Au/Au/SnSe₂ pressure sensor arrays.