Zinc oxide nanowires-based flexible pressure sensor

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Abstract
Embedding piezoelectric nanowires within a soft elastomer material should provide a superior pressure sensing transducer, exploiting the piezoelectric properties of the nanowire material while maintaining flexibility. Here, a flexible sensor has been fabricated on a Kapton substrate and has incorporated a layer of polydimethylsiloxane with embedded zinc oxide nanowires as the pressure sensing mechanism. In response to applied compressive pressure up to 127 kPa, the device has generated a voltage, between electrodes on either side of the nanowire/polydimethylsiloxane layer, with a sensitivity of 23.6 mV/kPa, which is 100 times greater than previously reported zinc oxide nanostructure-based flexible sensors.

1 | INTRODUCTION

Human-oriented technologies such as electronic and robotic skins, prosthetics, surgical robotic arms, rehabilitative devices and force-sensitive buttons on smartphones are becoming ubiquitous and part of daily life [1, 2]. Consequently, there is a requirement for improved pressure sensors [3, 4]. Current flexible pressure sensors need a transformative approach to design and fabrication in order to enhance functionality and overcome drawbacks such as integrability and limited wearability (flexible but not stretchable) and satisfy requirements such as near zero-power consumption and large pressure and spatial resolution. A promising area of research is the use of piezoelectric nanowires as the mechanism for transducing pressure into an electrical output [5].

This letter presents the utilisation of a flexible sensitive material that allows for dynamic pressure measurements. A simple fabrication process has been developed to embed piezoelectric zinc oxide (ZnO) nanowires into polydimethylsiloxane (PDMS), a flexible polymer material. A pressure applied to the device causes a deformation of the flexible PDMS and the ZnO nanowires, producing a voltage across the material.

2 | BACKGROUND

Previously reported research has demonstrated several transduction mechanisms for flexible pressure sensors. Piezoresistive sensing is achieved through measuring a change of resistance of a material, in response to an applied pressure [1, 6]. The measurement configuration can be as simple as connecting in series with a resistor and a DC source and there are a wide range of piezoresistive materials that can be used. However, piezoresistive sensors can suffer from hysteresis and the sensitivity can be dependent on the distribution of the pressure over the sensor area.

Sensors based on a capacitive transduction method [7] have a better degree of flexibility and stretchability but suffer from nonlinearity and need high frequency AC signal driving and electronics to transduce the change of capacitance into a corresponding pressure. Fabrication of capacitive sensors can be challenging, ensuring that the two conductive electrodes are separated by a suitable medium and distance.

For devices that incorporate a piezoelectric material, the applied pressure induces strain, which results in a change of electric field and voltage [8]. The quick response time allows for dynamic pressure measurements but their rigidity can pose reliability issues for flexible applications.

A promising solution to the drawbacks of existing measurement methods is the use of piezoelectric nanowires embedded in a flexible polymer. The synthesis and utilisation of piezoelectric nanowires has been demonstrated previously [9–14]. Rather than using a continuous piezoelectric layer, embedding an array of nanowires within a soft elastomer such as PDMS allows for the advantages of the piezoelectric material to be fully exploited, while eliminating the reliability issues associated with its rigidity.
Figure 1 shows a schematic of the fabrication process. First, a silicon wafer with a thickness of 380 μm has been diced into 30 mm × 30 mm square chips. Next, a 25 mm × 25 mm square sheet of Kapton (DuPont) with a thickness of 125 μm has been secured to a Si chip using adhesive Kapton tape. A 10 nm adhesion layer of titanium (Ti), followed by a 500 nm layer of aluminium (Al), an additional 10 nm adhesion layer of Ti and a 300 nm layer of silver (Ag) have been deposited using electron-beam evaporation. The Ag acts as a seed layer for the hydrothermal synthesis of ZnO nanowires. The Ag is masked with photoresist to expose a circle with a diameter of 10 mm, before the chip is immersed in a solution of zinc nitrate hexahydrate and hexamethylenetetramine mixed in equimolar concentration (80 mm) with deionised water (Figure 1(a)) [15]. The nanowires are defined into a circular pattern by removing the photoresist (Figure 1(b)). Next, PDMS (8 μm thick) is spin coated followed by sputtering of a top 500 nm Al electrode (Figure 1(c)). The Kapton substrate is removed from the Si and Al foil strips are connected, using Ag adhesive, to the top and bottom Al electrodes, before the entire device is encased in PDMS (Figure 1(d)), resulting in a device with a total thickness of 5 mm.

An optical image of a fabricated flexible pressure sensor is shown in Figure 2. As can be seen from the scanning electron microscopy (SEM) image in the inset of Figure 2, the ZnO nanowires are tightly packed with a cross sectional width of ≈1 μm. Through inspection with SEM, the height of the nanowires has been measured to be 5 μm.

4 | FINITE-ELEMENT METHOD SIMULATION

In order to determine the expected response of the sensor to pressure and characterise the experimental results, a finite-element method (FEM) simulation model has been created using CoventorWare. A 3D image of the meshed model is shown in Figure 3. The size and mesh density of the model has been chosen to optimise the simulation accuracy with a reasonable computation time. The model consists of a 250 × 250 μm square of Kapton with a thickness of 125 μm. A 500 nm layer of Al and a 300 nm layer of Ag have been stacked on top of the Kapton. Then, a 9 × 9 array of ZnO nanowires has been created on top of the Ag layer, as shown in Figure 3(b). Each wire has a height of 5 μm and a width and length of 2.5 μm, with a 2.5 μm spacing. Compared to the fabricated device, the nanowires in the simulation model have 2.5 times the width and spacing, but the same height, to allow sufficient freedom of movement to enable a successful simulation without excessive computation time. The nanowires have been encapsulated with a 100 μm layer of PDMS. The response of the model to an increasing pressure applied to the top of the PDMS has been simulated, with the piezoelectric voltage value from the nanowires extracted. The simulated response has been compared to the experimental response in the next section.

5 | EXPERIMENTAL PROCEDURE

The flexibility of the device has been demonstrated by positioning the device between the fixed chuck and the movable crosshead of a motorised test stand (Mark-10 ESM303). The crosshead has been moved to reduce the distance between two edges of the device from 30 mm down to 10 mm, reducing the bending radius down to 5 mm. When the crosshead is moved and the bending pressure is released, the device returns to its original shape, with no visible damage. The test has been
repeated 20 times in succession and the device continues to return to its original shape. For a final test, the bending radius has been reduced to below 1 mm, effectively folding the device in half, with no visible damage to the Kapton substrate or the encapsulating PDMS, demonstrating the device’s durability.

In order to characterise the electrical response of the device to a dynamic load, the top and bottom electrodes have been connected to a nanovoltmeter (Keithley 2182A) and the device has been subjected to increasing levels of compressive pressure.

The completed device has been positioned flat on the chuck of the motorised test stand and a flat, circular crosshead with a diameter of 12 mm has been moved down to apply a compressive pressure. The pressure has been maintained for 0.5 s and then the crosshead has been moved up so that the pressure is no longer being applied. Then, the process is repeated a further 20 times with a 0.5 s pause between each cycle. The voltage generated between the top and bottom electrodes has been recorded during the repeated application and release of the pressure. The testing has been performed for pressures from 6.35 kPa up to 127 kPa, and the mean voltage value for each pressure has been determined.

### 6 MEASUREMENT RESULTS AND DISCUSSION

Figure 4 shows the measured output voltage of the device as a function of the applied pressure. For each value of pressure, the box-plot shows the standard deviation of the measured voltage values. In addition, the whiskers above and below each box represent the maximum and minimum voltage measured. It can been seen that the output voltage increases as a function of the applied pressure.

In addition, Figure 4 shows the FEM simulated response of the model described in the previous section. It can be seen that the simulated result shows a linear relationship between pressure and the output voltage. When compared to the experimental result, the simulated response of the output voltage as a function of pressure shows a similar trend. The critical parameter of the FEM model is the $5 \mu m$ height of the nanowires, the same as for the fabricated device, resulting in reasonable agreement between the simulation and the experimental device output response, despite a 2.5 times larger nanowire width and spacing in the simulation.

Compared to previously reported research [11, 12] concerning flexible pressure sensors utilising ZnO nanostructures, which have demonstrated sensitivities up to 0.2 mV/kPa, the device that is presented here exhibits a 100 times greater sensitivity of 23.6 mV/kPa. It is thought that our use of PDMS, rather than polyimide, to encapsulate the nanowires allows for greater deformation to occur, thus maximising the generated voltage. The significantly lower Young’s modulus of PDMS ($\approx 1.7$ MPa) compared to polyimide ($\approx 2.5$ GPa) probably allows for a greater deformation in response to pressure.

Additional previous works report on pressure sensors where ZnO nanowires have been integrated into thin-film transistors [13] or a ZnO/poly(methyl methacrylate) composite material has been used as a dielectric in a capacitive sensing mechanism [14]. The devices both require a power supply to enable their output signal, either a change in source–drain current [13] or capacitance [14]. Our device requires no power supply, directly producing a voltage in response to pressure.
7 | CONCLUSION

A flexible pressure sensor that utilises piezoelectric ZnO nanowires embedded within PDMS has been fabricated and characterised. The device is based on a flexible Kapton substrate with the nanowires grown hydrothermally on an Ag seed layer before being encapsulated in a layer of PDMS. A bending radius of 5 mm has been demonstrated, with no mechanical damage to the structure. The device produces a voltage in response to an applied pressure with a sensitivity of 23.6 mV/kPa, ≈ 100 times greater than similar ZnO nanostructure-based pressure sensors. The performance of the device suggests that the utilisation of nanocomposite materials possesses a great deal of potential in flexible sensing applications.

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