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Hollow polydimethylsiloxane (PDMS) foam with a 3D interconnected network for highly sensitive capacitive pressure sensors

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Abstract

We propose a highly sensitive capacitive pressure sensor made of hollow polydimethylsiloxane (PDMS) foam with a three-dimensional network structure. The stiffness of the foam is adjusted by the viscosity of the PDMS solution. The fabricated PDMS-30 (PDMS 30 wt%) foam shows extremely high porosity (> 86%) approximately 19 times that of bare PDMS (PDMS 100 wt%) foam. Capacitive pressure sensors fabricated using the foam possess high sensitivity, good compressibility (up to 80% strain), and consistent output characteristics in a 2000-cycle test.

Keywords: Capacitive pressure sensor, Hollow PDMS foam, Porosity

Introduction

Over the last decade, flexible pressure sensors for a variety of wearable applications, such as electronic skin [1], human health monitoring [2, 3], and human–machine interfaces [4, 5], have received significant attention. Many researchers have concentrated on the development of various types of flexible and wearable pressure sensors, including piezoresistive [6, 7], capacitive [8–10], piezoelectric [11, 12], and triboelectric types [13, 14]. In particular, capacitive-type pressure sensors show significant advantages given their high sensitivity, low power consumption, simple design, and low hysteresis [15, 16]. A parallel-plate capacitive sensor is composed of a dielectric layer sandwiched between two conductive electrode plates for the detection of capacitance changes. With these sensors, the capacitance parameters change when external pressure is applied to the dielectric layer, with the result ultimately reflected in the change of the capacitance of the sensor. Specifically, the relative permittivity

ϵ_r and the distance between the electrodes d are the key factors affecting the performance capabilities of sensors such as those designed to measure pressure responsive sensitivity levels [17].

To ensure the fabrication of high-performance capacitive pressure sensors, recent studies have reported that microstructured or patterned polydimethylsiloxane (PDMS) dielectric layers represent the most effective means of improving the sensitivity of the capacitive-type sensors due to their excellent elasticity and biocompatibility [18, 19]. Pyramidal [20, 21], micro-pillar [22, 23], micro-wrinkle [24, 25], and microdome [26, 27] shapes created with elastic materials reportedly offer high sensitivity for use as electronic skin. However, the fabrication of a Si microstructured mold is complicated, with significant dependence on the equipment, multiple necessary steps, and high-cost manufacturing processes. Additionally, these sensors operate mainly at a low measurement level (< 10 kPa), which is insufficient for wearable systems in the medium–high pressure regime (10–100 kPa, suitable for object manipulation), as such systems must be capable of detecting changes [8].

For these reasons, the 3D monolithic porous PDMS dielectric layer has attracted much attention for use

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in capacitive-type pressure sensors [9]. Chhetry et al. reported a pressure sensor based on a dielectric hybrid sponge consisting of calcium copper titanate (CCTO), a material with very high dielectric permittivity, encased in polyurethane (PU) for detection in low-pressure regime (<1.6 kPa) [28]. Pruvost et al. also suggested a PDMS foam decorated with carbon black particles for use in the fabrication of highly sensitive capacitive pressure sensors exceeding 35 kPa^{-1} for pressures of <0.2 kPa [29]. These studies focused on improving performance capabilities via the use of conductive nanomaterials while maintaining the porosity of the sponge. However, aggregation and agglomeration cause low production reliability of composites of nanomaterials and elastomers, and the methods above cannot regulate or tune sensor sensitivity levels. The easiest and fastest way to tune a sensor without conductive nanomaterials is to adjust its porosity. Thus, many studies have focused on high sensitivity and wide sensing ranges when applying a porous elastomer to a capacitive pressure sensor. Kang et al. suggested a capacitive pressure sensor with a porous structure consisting of a polydimethylsiloxane (PDMS) thin film dielectric layer. The morphology of the porous structured dielectric layer could be controlled by changing the pore sizes [30]. Jung et al. showed that a PDMS and microsphere composite could be applied to a capacitive pressure sensor by maximizing the porosity of microspheres via the effect of high compressibility and an enhanced piezocapacitive effect [9]. Li et al. also reported a pressure sensor based on a dielectric layer of PDMS with uniformly distributed micropores which displayed high elasticity, a wide pressure-sensing range (>200 kPa), and high sensitivity (0.023 kPa^{-1}) [31].

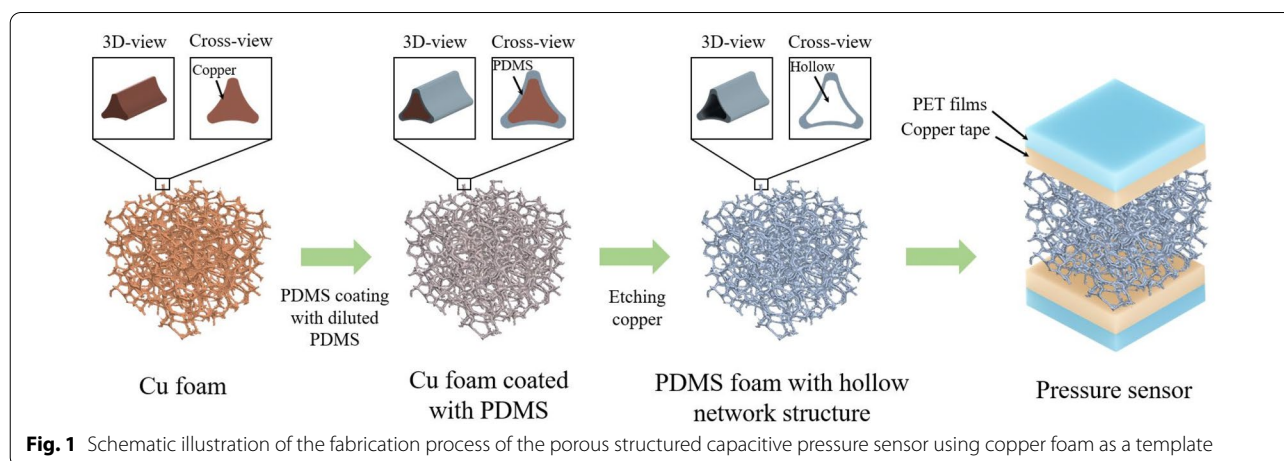
In this report, we propose a facile approach by which to adjust the porosity of PDMS foam by controlling the viscosity of the PDMS immersion solution. Due to the

synergy effect of the high elasticity of the PDMS elastomer and the highly porous 3D micro-hollow network structure, the as-prepared highly porous PDMS dielectric layer showed excellent mechanical resilience, extremely high compressibility, and stable cyclic performance. These flexible pressure sensors are also capable of not only detecting low pressure levels but also enabling real-time human motion sensing applications.

Preparation of the hollow PDMS foam and the pressure sensor

Figure 1 shows a schematic of the fabrication process of the PDMS foam for the capacitive pressure sensor. Commercial copper foam (supplied by ITASCO) with porosity of approximately 95% was used as template. First, a PDMS precursor (Sylgard 184 Silicone Elastomer, Dow Corning) consisting of a mixture of a resin and a hardener at a 5:1 ratio was prepared and placed in a vacuum chamber for 20 min to remove bubbles. In this case, a PDMS diluent (OS-10, Sylgard 184, Dow Corning Corp., USA) is used to adjust the porosity of the PDMS foam. PDMS solution samples of 30 and 50 wt% in terms of the PDMS diluent were created by adding set amounts of the PDMS diluent to the PDMS precursor mixture, followed by ultrasonication for 30 min. Here, PDMS foam samples with different porosity levels are denoted as PDMS- x , where x denotes the weight percentage (wt%) of PDMS in the immersion solution. The porosity of the PDMS dielectric layer in the fabricated pressure sensors was controlled by changing the weight percentage of PDMS in the immersion solution.

In order to remove impurities remaining on the copper surface, the copper foam is rinsed with acetone, ethanol, and deionized water. After drying for 30 min in an oven at 60°C , the copper foam was then soaked in a diluted PDMS solution (30, 50 wt%) for an hour to ensure a



conformal coating, after which it was dried at 90 °C for 1 h. For the bare PDMS foam, the copper foam was then immersed into the PDMS precursor mixture for 1 h and cured at 90 °C for 3 h. Finally, the sacrificial copper foam template was removed by a wet etching method. The PDMS-coated copper foam was then positioned such that it floated on the solutions of acetic acid, hydrogen peroxide, and deionized (DI) water at a ratio of 1:1:5 for 12 h to etch away the copper foam. After completely etching the copper, the PDMS foam was soaked in deionized (DI) water for 6 h to remove the acid residue. Subsequently, the PDMS foam samples were dried at room temperature. The fabrication process finished with the formation of the top and bottom electrodes. Polyethylene terephthalate (PET) films (Fine chemical Industry, Korea) attachable with double-sided bonding copper conductive tape (DTS-272, Ducksunhitech Corp., Korea) were attached onto the top and bottom surfaces of the fabricated PDMS foam.

Characterization

To characterize the morphology of the porous PDMS foam, field emission scanning electron microscopy (FE-SEM, SUPRA 25, Zeiss, Germany) was used. To measure the compressive stress–strain curves at various compressive strain levels, a universal testing machine (JSV-H1000, Japan) was employed to characterize the performance with a load cell (HF-1, maximum load 10 N, load resolution 0.001 N). The fabricated PDMS foam samples were compressed at a speed of 10% strain

min⁻¹ for a mechanical evaluation. The capacitance of the pressure sensors was measured using an LCR meter (Hioki-3536, Hioki, Japan) at 200 kHz with 1 V of bias. A universal testing machine (JSV-H1000, Japan) was used to apply the pressure and a load cell (HF-1, maximum load 10 N, load resolution 0.001 N) measured the applied pressure values. All sensor evaluations were conducted by connecting the LCR meter to a computer for real-time measurements.

Results and discussion

To confirm the effect of the diluent on the porosity of the PDMS foam structure, FE-SEM was utilized on various locations of the samples. The porosity of the fabricated PDMS foams is calculated from the density of the PDMS foam (ρ_{foam}) and the bulk PDMS density (ρ_{bulk}) using Eq. (1):

$$f = \left(1 - \frac{\rho_{\text{foam}}}{\rho_{\text{bulk}}} \right) \times 100(\%), \quad (1)$$

where $\rho_{\text{foam}} = 1100 \text{ kg/m}^3$ [31]. By varying the weight concentration of PDMS in the immersion solution, the PDMS-30 foam sample showed porosity that exceeded 86%, i.e., nearly 19 times higher than that of the bare PDMS foam (Fig. 2a). It was also found that a lower PDMS weight concentration of the immersion solution led to higher porosity of the PDMS foam. This occurred because the higher PDMS diluent ($\sim 10 \text{ cP}$) weight concentration reduces the viscosity of the immersion solution and the capillary force during the micro-molding

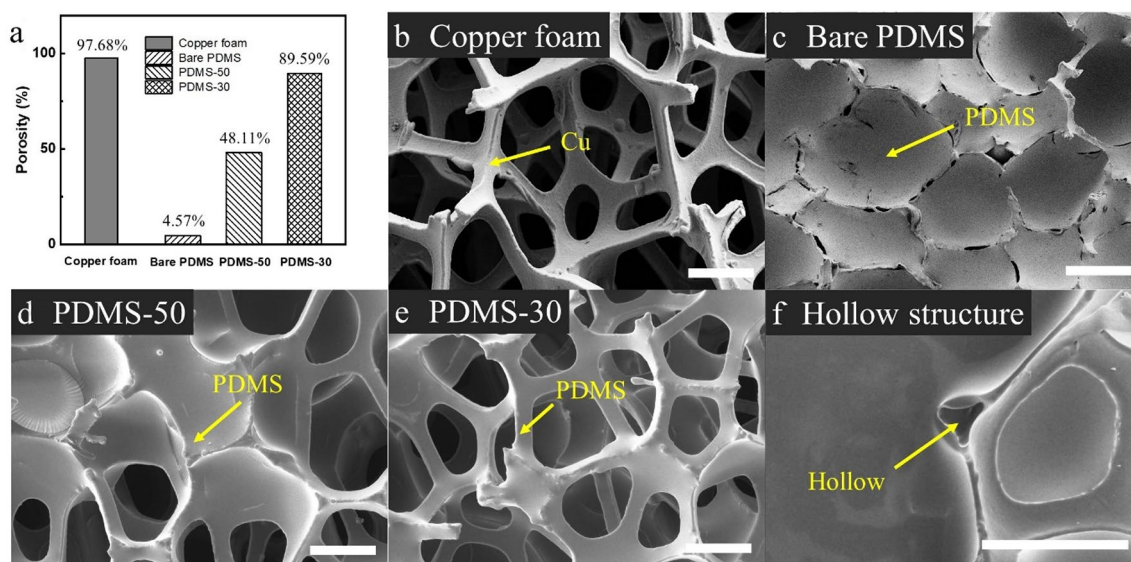


Fig. 2 a Porosity of the copper foam and the fabricated pressure sensors. SEM images are shown of b the copper foam and the pressure sensor based on c bare PDMS, d PDMS-50, e PDMS-30, and f the hollow structure of PDMS-30

process is inversely proportional to the viscosity [32]. Therefore, at higher weight concentrations of PDMS in the immersion solution, more pores of the copper foam (Fig. 2b) remained filled with PDMS until the elastomer was cured owing to the increased capillary force (Figs. 2c, d). In contrast, most of the pores were empty when diluted PDMS solutions were used (Fig. 2e). In the PDMS-30 case, only the coated PDMS remained to form a hollow network structure after the copper foam was etched away (Fig. 2f).

To evaluate the mechanical behaviors of the PDMS foams, uniaxial compression tests were conducted. Figure 3a shows the stress–strain curves for the PDMS foam samples with different concentrations of PDMS in the immersion solution at 80% compressive strain. To quantify the hysteresis of the mechanical behavior of PDMS foam, the hysteresis error of the stress can be calculated as follow [34]:

$$\gamma_H = \pm \frac{\Delta H_{\max}}{Y_{FS}} \times 100(\%), \quad (2)$$

$$\Delta H_{\max} = \text{MAX}|\varepsilon_L(s,t) - \varepsilon_U(s,t)| \quad (3)$$

Here, ΔH_{\max} represents the largest deviation between the loading and unloading. $\varepsilon_L(s, t)$ is the compressive strain of the PDMS foam under loading pressure and $\varepsilon_U(s, t)$ is the resistance of the PDMS foam under the unloading pressure. The hysteresis errors of the compressive strain of the bare PDMS, PDMS-50, and PDMS-30 samples were $\pm 13.29\%$, $\pm 12.14\%$, and $\pm 10.17\%$, indicating better recovery performance of the diluted PDMS foams. Additionally, It can be observed that as the concentration of the PDMS was increased, the maximum compressive stress increased. In the bare PDMS case, pressure of approximately 758.96 kPa was required to reach 80% strain, whereas the PDMS-30 required pressure of only 40.24 kPa for the same level of strain. These results indicate that the stiffness of the porous PDMS structure coated onto the copper foam with the diluted PDMS is reduced, making the structure easily compressible with pressure. The elastic properties of the PDMS-30 sample with the highest porosity were further characterized to determine the extent of its mechanical resilience against compression.

Figure 3b shows the stress–strain curves after four compression cycles with strain amplitudes of 20%, 40%, 60%, and 80% in sequence. These results indicate that each loading curve traces the maximum stress of the preceding curve. Furthermore, the stress–strain curves of the PDMS-30 sample were measured with different

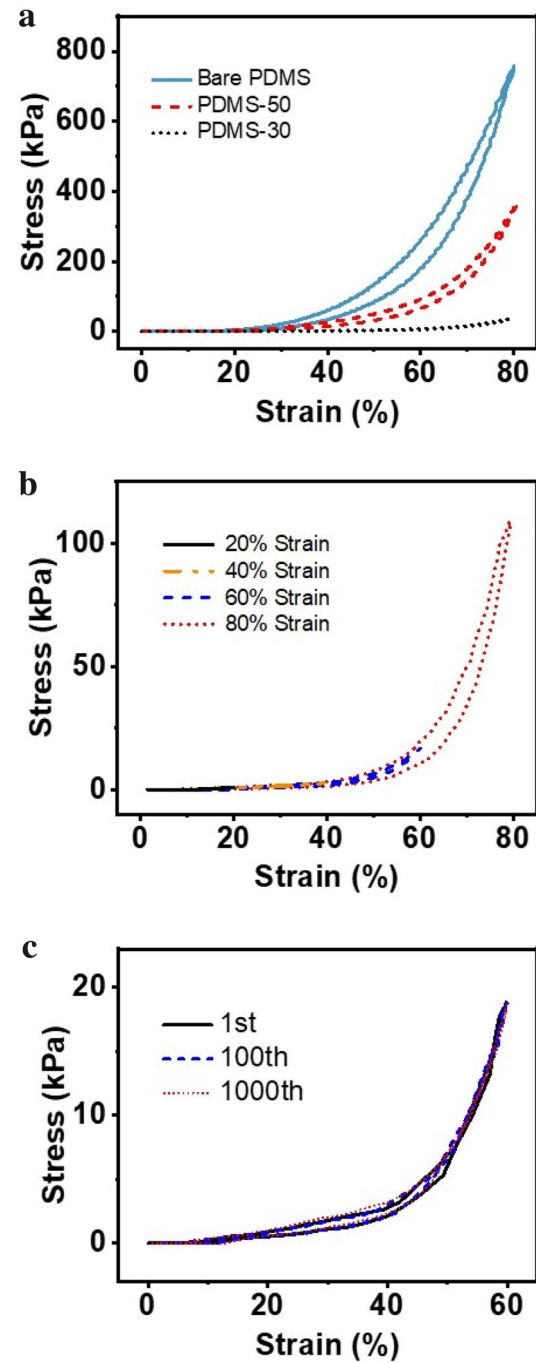


Fig. 3 **a** Stress–strain curves of the pressure sensor based on various weight concentrations of bare PDMS (blue solid line), in this case 50 wt% (red dash line) and 30 wt% (black dot line) at 80% compressive strain. **b** Sequential loading–unloading cycles of PDMS-30 foam under compressive strain ranging from 20 to 80%. **c** Cyclic compression tests of the PDMS-30 foam sample at 60% compressive strain

consecutive loading–unloading cycles, as shown in Fig. 3c. These results showed very low hysteresis after 1,000 cycles. This remarkable mechanical behavior stems from the highly elastic nature of the PDMS and the highly porous structure of the fabricated foam. The improved elasticity can be explained by the higher degree of porosity, which offers more free space for more intensive buckling and bending of the frame pillars of the foam.

Figure 4a shows the relative changes of the capacitance ($\Delta C/C_0$) of the pressure sensors as a function of the compressive strain in the pressure range of 0 to 100 kPa. There is a tendency for the relative change of the capacitance to increase as the porosity is increased. At an

applied pressure level of 20 kPa, whereas the bare PDMS foam showed relative capacitance of only 0.21, this value for the PDMS-30 foam was 0.67. As shown in Fig. 3a, the mechanical strength of a high-porosity sample is higher than that of a low-porosity sample. Therefore, the PDMS-30 foam is more easily compressed than the bare PDMS foam. The relatively high compression results in a higher relative change of the capacitance. Moreover, changes in the relative permittivity may contribute significantly to the sensitivity of these structures [17]. When structured films are compressed, the air volume is reduced, resulting in an increase in the effective dielectric constant given that the dielectric constant of air ($\epsilon_{air} \sim 1$) is lower than that of PDMS elastomer ($\epsilon_{PDMS} \sim 2.69$ [30]). Accordingly,

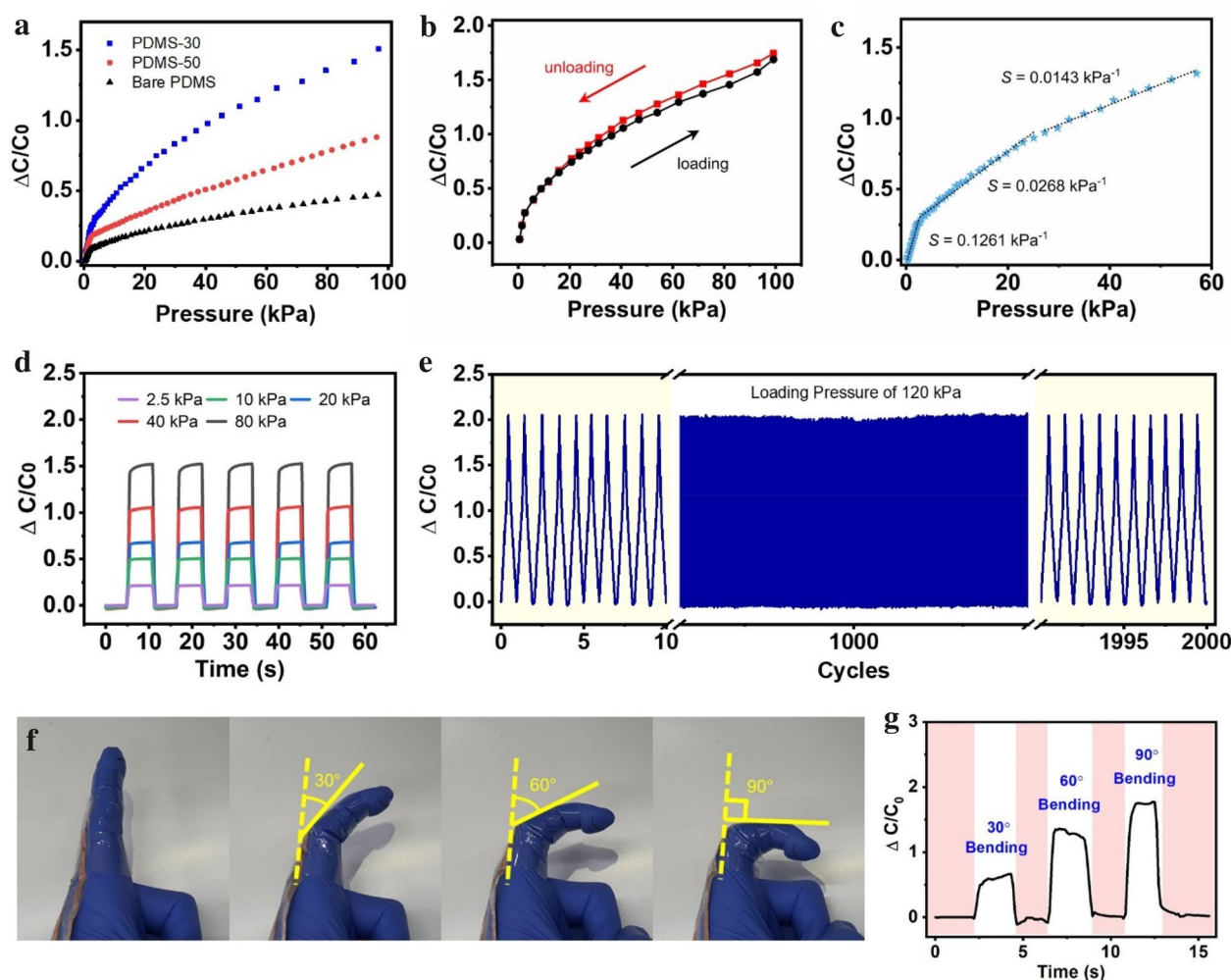


Fig. 4 **a** Pressure-response (relative capacitance) curves as function of the applied pressure for three weight concentrations of PDMS: 30 wt% (blue squares), 50 wt% (red circles), and the bare PDMS sample (black triangles). **b** Hysteresis characteristics of the PDMS-30-based pressure sensor. **c** Sensitivity measurements S in three different pressure ranges (PDMS-30). **d** Capacitance responses of the PDMS-30-based pressure sensor under various applied pressures. **e** Working stability and durability outcomes of the PDMS-30-based pressure sensor after 2000 cycles under applied pressure of 120 kPa. **f** Images of the index finger for three different bending angles. **g** Capacitance responses of the bending of the index finger

higher porosity causes a greater change in the relative permittivity, leading to an increase in the sensitivity of the pressure sensor.

To investigate the hysteresis characteristics of the sensor, a loading–unloading cycle test was conducted (Fig. 4b). Figure 4c exhibits the different sensitivities of the pressure sensor based on PDMS-30, showing three different linear regions (0–2.5, 2.5–25, and 25–60 kPa) depending on the pressure level. The maximum sensitivity is found to be 0.1261 kPa^{-1} in the 0–2.5 kPa (region 1) range. Sensitivity values of 0.0268 kPa^{-1} and 0.0143 kPa^{-1} were measured in the 2.5–25 kPa (region 2) and 25–60 kPa ranges (region 3), respectively. There is a reduction in the sensitivity of the pressure sensor as the applied pressure increases; this can be attributed to the effective relative dielectric constant. The effective relative dielectric constant, ϵ_r , can be estimated by the equation $\epsilon_r = \epsilon_{air} P_{air} + \epsilon_{PDMS} P_{PDMS}$, where P_{air} and P_{PDMS} refer the volume proportion of air and PDMS in the porous PDMS dielectric layer, respectively [30]. When pressure is applied to the pressure sensor, the proportion of air, which has relatively low permittivity, decreases, resulting in increases of the effective permittivity of the dielectric layer and the capacitance of the pressure sensor. In the high-pressure region (region 2, 3), because the pores were already almost fully compressed, similar to unstructured film, the dielectric layer had a lower effective elasticity. The sensitivity in these regions is therefore considerably lower than that in the low pressure region (region 1).

Figure 4d shows the stable capacitance responses of the pressure sensor under various applied pressures, indicating that the pressure sensor returned to its initial state after the external pressure was removed. At an applied pressure of 2.5 kPa, the 10–90% rise time (t_r) and 90%–10% fall time (t_d) were measured and found to be 172 ms and 145 ms, respectively. To confirm the durability and stability of the sensor for long-term use, the capacitance response to an applied pressure of 120 kPa for 2,000 cycles was measured (Fig. 4e). During the initial ten cycles (cycles 1–10), the average relative capacitance value and the deviation of the sensors were found to be 2.052 and 0.89%, respectively. Correspondingly, these values were 2.049 and 0.78% during the middle cycles (cycles 995–1,004) and 2.048 and 0.67% during the final ten cycles (cycles 1,991–2,000). The PDMS-30 pressure sensor showed a nearly identical average relative capacitance value, and the standard deviations were lower than 1% during the 2,000-cycle test. For convenient use in human body motion monitoring applications, the capacitive pressure sensor based on PDMS-30 was adhered via Scotch tape onto the index finger to take measurements during a finger bending test. As shown in Fig. 4f,

the index finger was bent at 30, 60 and 90 degrees, and the capacitance was measured in real time by the pressure sensor. The pressure-sensing signals are presented in Fig. 4g, which are clearly in good agreement with the variation with the motion of the finger. Therefore, the sensor shows strong potential to detect human body motions.

Conclusion

In summary, we developed facile capacitive pressure sensors based on hollow PDMS foam with a 3D interconnected network structure. The fabricated PDMS foam displayed remarkable mechanical resilience and extremely high compressibility with good cyclic performance due to the synergistic effect of the increased porosity and high elasticity of the PDMS. The pressure sensor based on PDMS-30 showed higher sensitivity than the bare PDMS sensor given the reduced stiffness and effective relative permittivity at identical levels of pressure and strain. Furthermore, the capacitive pressure sensor exhibited low hysteresis, a wide pressure range, and high working durability at 120 kPa for 2,000 repeating cycles. Finally, we demonstrate that our designed pressure sensors could be applied to detect human body motion signals such as finger bending in real time. We believe that this highly porous capacitive pressure sensor can achieve real-time monitoring in wide pressure ranges for use in a variety of wearable applications, such as human motion detection, human–machine interaction, and as e-skin for robotic applications.

Abbreviations

PDMS: Polydimethylsiloxane; e-skin: Electronic skin; ϵ_r : The relative permittivity; d : the distance between two electrodes; CCTO: Calcium Copper titanate; PU: Polyurethane; wt%: Weight percent; DI: Deionized; PETs: Polyethyleneterephthalate films; FE-SEM: Field emission scanning electron microscopy; ΔC : Relative change of the capacitance; C_0 : The capacitance without applied pressure; S : The sensitivity; p : The applied pressure.

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Authors' contributions

DHK and YJ developed the idea. KKJ and MGS carried out fabrication and literature search. DHK, DMP, and YJ contributed to measurement. DHK, JY, HJT, and DHK(Dong Hwa Kwak) drafted the manuscript. JSK supervised the research and reviewed the manuscript. The manuscript was written through contributions from all authors. All authors read and approved the final manuscript.

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Availability of data and materials

All data generated or analyzed during this study are included in this published article.

Competing interests

The authors declare no competing financial interests.

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