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Research paper

Fabrication of highly sensitive capacitive pressure sensors with porous PDMS dielectric layer via microwave treatment

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ABSTRACT

This work aims to present a flexible, highly sensitive, capacitive pressure sensor based on an elastomeric dielectric layer with uniformly distributed micro-pores. The porous dielectric layer was simply produced by microwave treatment of polydimethylsiloxane (PDMS) mixed with a sacrificial solvent within a few minutes. The pressure sensor using a porous PDMS dielectric layer fabricated with 40 wt% sacrificial solvent had a sensitivity of 0.813 kPa^{-1}, which is 11 times higher than that of the sensor using a non-porous dielectric layer. To further improve the sensitivity, low-cost commercial glass was used as a mold to form microstructures on the surface of the porous dielectric layer. Due to the enhanced deformability, high sensitivity of 1.43 kPa^{-1} and a fast response time of 70 ms was achieved. The performances of a sensor array as well as finger sensors were also demonstrated by placing light-weight objects on the array and holding a plastic cup. It would be expected that the proposed sensors could be utilized in the application of artificial skins or soft robotics.

1. Introduction

Flexible pressure sensors are receiving much attention these days due to their wide applications in electronic skin, flexible touch sensors, soft robotics, and wearable electronics. Many studies have been conducted for their use as a medical instrument or to replace the human skin [1–6]. Among the various types of pressure sensors, such as capacitive [7–15], piezo-resistive [16–20], and piezo-electric types [21–23], the capacitive type pressure sensor has been attracting the most attention because of its simple structure, low power consumption, and excellent stability.

The main challenge of capacitive pressure sensors is to achieve the high sensitivity of the sensor toward very small signals like bio-signals such as pulses, textile sensing, and blood pressure. Capacitive pressure sensors work by measuring the variation in the capacitance value in response to applied external pressure. Since the capacitance value varies with changes in the thickness of the dielectric layer under pressure, high deformability of the dielectric layer is required to realize the high sensitivity of the pressure sensor. Elastomers such as polydimethylsiloxane (PDMS) have been studied extensively due to their low Young's modulus, inherent transparency, stability to temperature and humidity, environmental friendliness and excellent permeability [1,7,8,10,12,14,15,17].

Recently, various attempts have been made to improve the sensitivity of capacitive pressure sensors using structured dielectric layers. Some researchers have improved the deformability of the dielectric layer by forming microstructures on the surface of the layer [11,14]. They achieved relatively high sensitivity, but the complicated and expensive photolithography processes were used to fabricate microstructures. Another method to improve sensitivity is to form porous structures inside the dielectric layer [7,10,15]. In this method, the porous dielectric layers were fabricated by curing PDMS with sugar, salt or poly(methyl methacrylate) (PMMA) particles and then dissolving it in water or acetic acid. However, this method requires complex manufacturing processes and large amounts of time up to 24 h. In addition, the sensitivities of the sensors were not sufficiently high. Therefore, a simple but efficient way to fabricate porous dielectric layers is still necessary for fabricating highly sensitive sensors.

In our previous work, we introduced a process to fabricate porous PDMS using microwave irradiation [24]. Microwave treatment of the PDMS which is mixed with a sacrificial solvent causes the molecules of the solvent to rapidly heat up and cure the PDMS simultaneously, resulting in the formation of porous structures. We have also found that the pore size and the porosity can be controlled based on the boiling point of the sacrificial solvent.

In this study, we introduced a highly sensitive capacitive pressure sensor with an easily-fabricated porous PDMS dielectric layer. Microwave treatment was used as a simple and cost-effective

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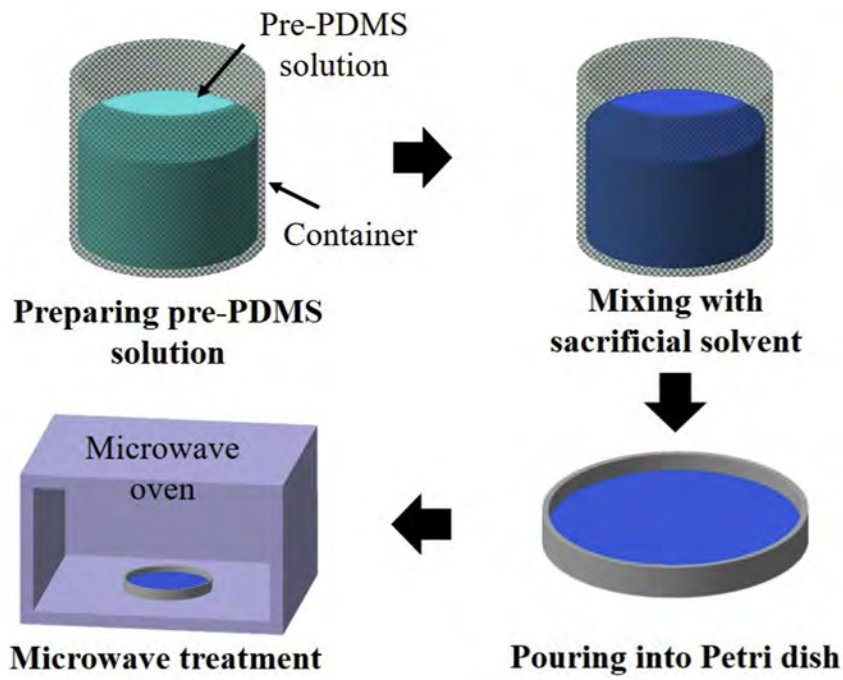

Fig. 1. Schematic of the fabrication process of a porous PDMS film.

fabrication method for producing porous dielectric layers, and the effects of the sacrificial solvent ratio on pore formation and sensor sensitivity were investigated. The sensor with a porous dielectric layer showed higher sensitivity than that using a pore-free dielectric layer, and the sensitivity was further improved when microstructures were formed using a cheap and commercially-available glass mold on the surface of the layer. A sensor array and finger sensors were also fabricated and characterized as an application of the proposed sensors.

2. Experimental details

2.1. Fabrication of dielectric layer

Fig. 1 shows the fabrication process of a porous dielectric layer using microwave treatment. PDMS (Sylgard 184, Dow Chemical Co.) was used as a dielectric material, and perfluorocarbon (FC-43, 3 M) was used as a sacrificial solvent. PDMS prepolymer and curing agent were mixed at a ratio of 20:1, and the sacrificial solvent was dispersed in pre-PDMS solution at a concentration of 0 to 50 wt% in intervals of 10 wt%. The dispersion was mechanically mixed for 6 min at 2000 rpm using a homo-mixer (ARE-310, Thinky Co.). The dispersion of 4 ml was poured into a petri dish, degassed in vacuum for 10 min to remove any remaining air and spin-coated at 200 rpm for one minute. The fabrication process of the porous PDMS (P-PDMS) was completed after microwave treatment for 3 min using a household microwave oven. The microwave oven was used in the fume hood to prevent hazardous gases produced during the treatment of FC-43. The fabricated P-PDMS film had a thickness of 400 μm.

A commercially available opaque glass plate was used as a mold to create microstructures on the surface of the P-PDMS film. The dispersion of 4 ml of PDMS and the sacrificial solvent was poured on the glass mold placed on the bottom of a petri dish and spin-coated at 450 rpm for 30 s. The subsequent process was the same as the previously mentioned process for the fabrication of the porous dielectric layer.

2.2. Fabrication of pressure sensors and sensor array

The capacitive pressure sensors were fabricated based on the P-PDMS dielectric layer. An aluminum (Al) fabric was used for the electrode, and it was bonded with a PDMS substrate using a silicone epoxy. The P-PDMS dielectric layer was placed between two electrodes and bonded using a PDMS solution, and it was cured at 70 °C for 4 h for the perfect bonding of each layer. Overlapped area of the two electrodes was 10 mm by 10 mm. The top and bottom PDMS substrates bonded with Al electrode were 300 μm thick, respectively, so the total thickness of the fabricated sensor was 1 mm. For more accurate measurement of the electrical characteristics, copper wires were connected to the electrodes using silver epoxy.

The sensor array was fabricated in the same manner as above, in which Al electrodes having a width of 3 mm were bonded with the PDMS substrate. The distance between Al electrodes was set to 3 mm, and four electrodes on the top and bottom surfaces were placed perpendicular to each other.

2.3. Characterization and evaluation

Pore formation of the dielectric layer with different sacrificial solvent ratios was examined using cross-sectional images from a field emission-scanning electron microscope (FE-SEM; MIRA3, TESCAN). Changes in capacitance values under external pressure were measured using an LCR meter (IM-3523, HIOKI) to investigate the effects of the porous structures and microstructures on the sensitivity of the sensor. The external load was applied using an in-house *Z*-axis stage, and the applied pressure on a sensor was estimated with the help of a load cell (UMM−K20, Dacell Co.) installed at the stage. During the pressure measurement, the Z-axis stage was moved at a speed of 10 μm/s. We also evaluated the reaction of the sensor by tapping a table and grabbing a cup with sensors attached to the fingers.

3. Results and discussion

3.1. Micro-pore formation in a dielectric layer

The difference in porous structures inside the microwave-irradiated PDMS film was investigated as a function of the sacrificial solvent ratio using FE-SEM images. As shown in Fig. 2, many micro-pores were uniformly generated in the PDMS due to the higher boiling point of the sacrificial solvent [24]. When a solvent with low boiling point is used,

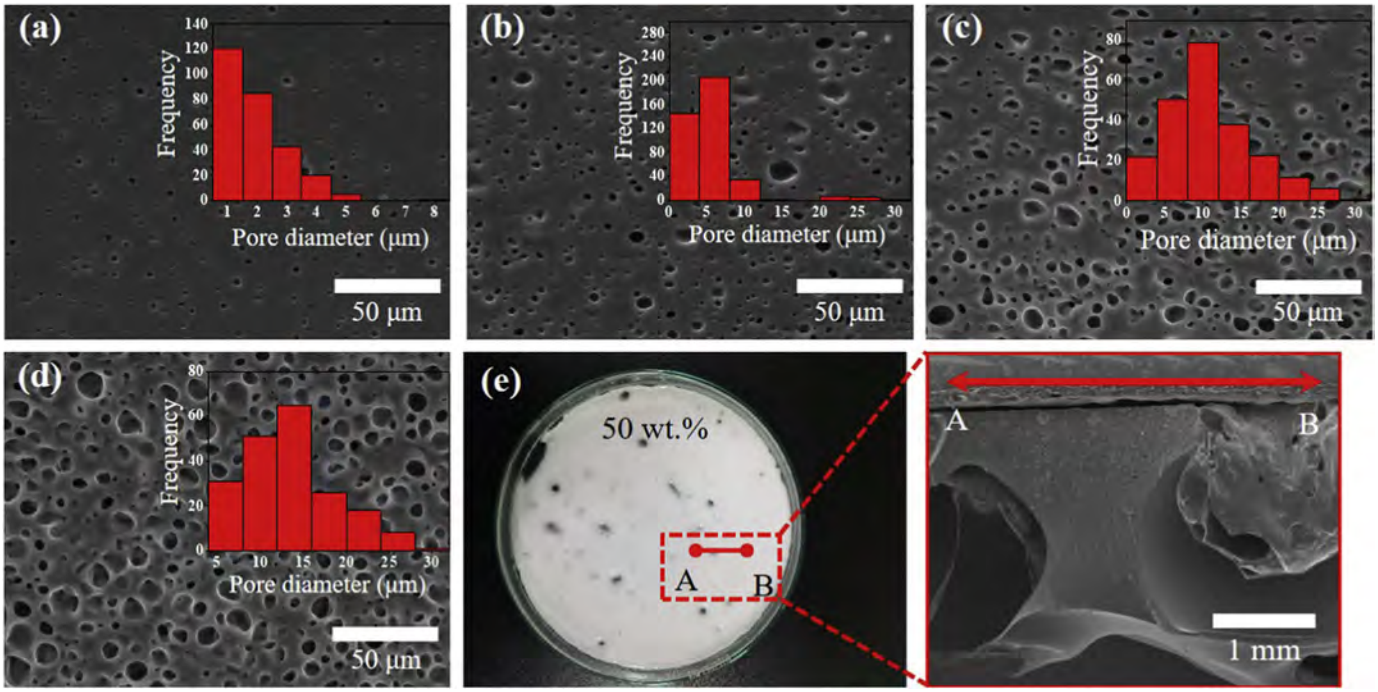

Fig. 2. Cross-sectional SEM images of P-PDMS and pore size distribution with a sacrificial solvent ratio of (a) 10 wt%, (b) 20 wt%, (c) 30 wt%, and (d) 40 wt%. (e) A non-uniform film fabricated with a sacrificial solvent ratio of 50 wt%. SEM image in the red box shows the cross-sectional view between point A and B. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

the excessive and rapid expansion of the gas occurs due to extreme transition from liquid to a gas phase. In this case, the bulky solutions aggregate together to form large pores before the PDMS is cured, resulting in fabrication of a sponge-like dielectric layer. On the other hand, when the boiling point of a sacrificial solvent is relatively high, the pores do not clump together. Small pores can be formed since the curing process and the phase change of the sacrificial solvent simultaneously progress. Phase-changed solvent would be trapped in cured PDMS layer and micro-pores are fabricated. The curing of PDMS also occurred rapidly during the evaporation of the solvent due to internally generated heat from microwave irradiation.

The pore size and porosity increased with increasing the ratio of the sacrificial solvent. Pores of the adjacent solvent regions are more likely to coalesce as more solvent is added during the fabrication of the dielectric layer. The pore size and porosity were nearly proportional to the solvent ratio, indicating that they can be easily controlled. However, when the portion of the solvent was higher than 50 wt%, the solvent and PDMS were not completely dispersed, resulting in the solvent macroscopically separating from the PDMS. A non-uniform film was produced when microwave was irradiated to this sample as shown in Fig. 2(e). Therefore, we conducted further experiments up to 40 wt% of the sacrificial solvent.

3.2. Pressure sensors with a porous dielectric layer

We investigated the effects of the solvent ratio on the sensitivity of the pressure sensors. Fig. 3(a) and (b) shows the structure and the photograph of a fabricated sensor, respectively. Capacitance changes under external pressure were measured using an LCR meter to characterize the capacitive pressure sensors. Fig. 3(c) shows the capacitance change ratio ($\Delta C/C_0$) with respect to the applied external pressure. The sensitivity of a capacitive pressure sensor is defined as the slope of this curve, i.e., the capacitance change ratio divided by the pressure change. As can be seen from the graph, $\Delta C/C_0$ as a function of pressure became larger with increasing the solvent ratio, and all the curves were divided into two regions like a bi-linear function.

This can be explained by three reasons. The main reason is due to the change in the shapes of pores inside the dielectric layer under pressure (Fig. 3(d)). When the pressure is applied to the sensor, the pores begin to deform from their original shapes. Since Young's modulus (E) of the pores is negligible, E value of the original, undeformed P-PDMS film is much smaller than that of solid PDMS. Therefore, it is easier to deform the P-PDMS film even at a lower pressure, leading to a larger change in capacitance. However, as the pressure increases, the pore's deformation becomes severer, and its shape becomes flatter. In this case, the deformed P-PDMS films would have the almost same E value to the solid PDMS film. As a result, the variation in the capacitance value is also reduced under higher pressure. Increased pore size and porosity of P-PDMS make this phenomenon more precise, which results in the most significant variation of $\Delta C/C_0$ at the solvent ratio of 40 wt% under both lower and higher pressure regions. Similarly, as the sensor is pressurized, the portion of the air, which has low dielectric constant, decreases and thus the effective permittivity of the dielectric layer increases [13,25]. When the applied force reaches a certain value, the pores in the dielectric layer would be closed, and the effective dielectric constant no longer changes. The capacitance change ratio should be much lower under this circumstance. Another reason is the viscoelastic behavior of the PDMS dielectric layer [26–28]. Viscoelasticity is the time-dependent elastic behavior of materials. Shear modulus (G') and loss factor (tan(δ)) of viscoelastic materials lead to non-linear curves in terms of time. Especially, G' and tan(δ) of PDMS shows distinct bi-linear curves, and this viscoelasticity affects the compressive strain of the PDMS dielectric layer, resulting in bi-linear capacitance change ratio ($\Delta C/C0$) under applied external pressure of the proposed sensors.

As expected, the sensitivity of the pressure sensor increased as the solvent ratio increased (Fig. 3(e)). The sensitivities in the low-pressure region ($<$ 0.2 kPa) were much higher than those in the high-pressure region ($>$ 0.2 kPa). In the low-pressure region, the sensitivities were 0.0725, 0.1094, 0.139, 0.269, and 0.813 kPa^{-1} with the solvent ratios of 0, 10, 20, 30, and 40 wt%, respectively. In other words, larger pores and higher porosity could increase the deformability of the dielectric

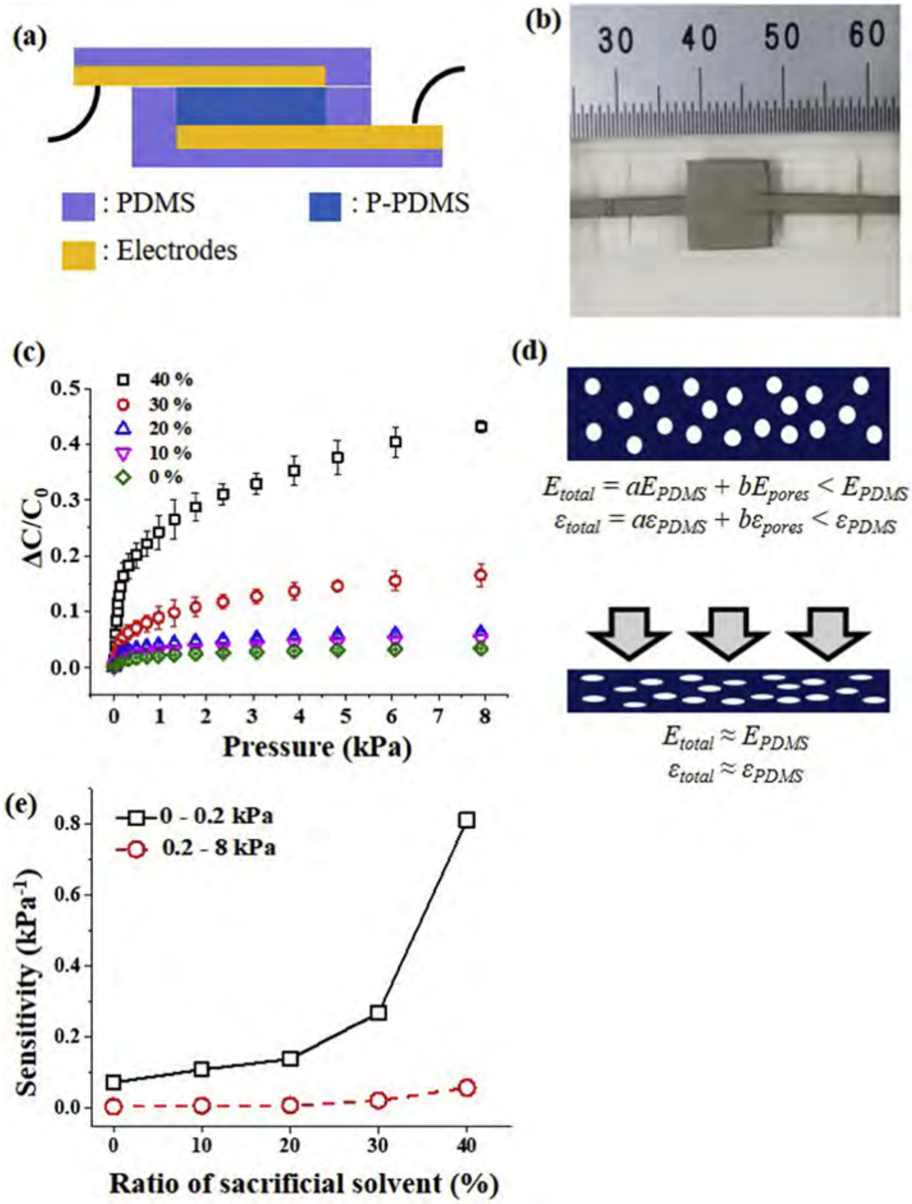

Fig. 3. (a) The structure and (b) a photograph of a pressure sensor. (c) Capacitance change ratio of the pressure sensor with respect to the applied external pressure. The legend shows the ratio of the sacrificial solvent, and error bars indicate standard deviation of three samples. (d) Change in Young's modulus and effective dielectric constant of the dielectric layer in the P-PDMS film under pressure loading. (e) Sensitivities as a function of the sacrificial solvent ratio at the pressures of 0 kPa – 0.2 kPa and 0.2 kPa – 8 kPa.

layer, resulting in higher sensitivity. It should also be noted that the pressure sensor with a sacrificial solvent ratio of 40 wt% showed about 11.2 times higher sensitivity than that with the solid PDMS. Even though the sensitivity in the high-pressure region had a smaller value, it also tended to increase with increasing the solvent ratio.

3.3. *Effect of surface microstructure on pressure sensor*

The sensitivity can be further improved by forming microstructures on the surface of the dielectric layer. In order to easily and cheaply create the surface microstructure for P-PDMS, we used a commercial opaque glass plate as a mold. This glass plate costs only $5 for a 300 mm × 300 mm piece, and is readily available and usually used for office partitions. Fig. 4(a) shows a photograph of the opaque glass mold and an SEM image of its surface. Each pattern had a small hill shape with a square bottom. The length of a side was 500 µm, and the depth was 100 µm. Fig. 4(b) shows the surface of P-PDMS film with the sacrificial solvent ratio of 40 wt% after pattern transfer. The patterns of the glass mold were well-transferred on the surface of the P-PDMS film. The porous structures were also well-fabricated with a uniformly distributed pores (inset image in Fig. 4(b)).

The sensitivity of the fabricated sensor using P-PDMS with surface microstructures was 1.43 kPa^{-1} and 0.102 kPa^{-1} in the low and high-pressure regions, respectively (Fig. 4(c)). The sensor with a microstructured P-PDMS dielectric layer exhibited a sensitivity 1.7 times higher than that with only porous structures. This result is an excellent achievement considering the fabrication simplicity and cost. More delicate surface structures such as pyramids also showed reasonable enhancement [14], but they require a complicated manufacturing process and a considerably higher cost. In contrast, the opaque glass mold used in this work can enhance the sensitivity at an extremely low cost. The proposed two-step fabrication method, P-PDMS plus low-cost surface microstructure, would help to obtain a highly sensitive pressure sensor in a simple, fast, and cost-effective way. We also checked the stability of

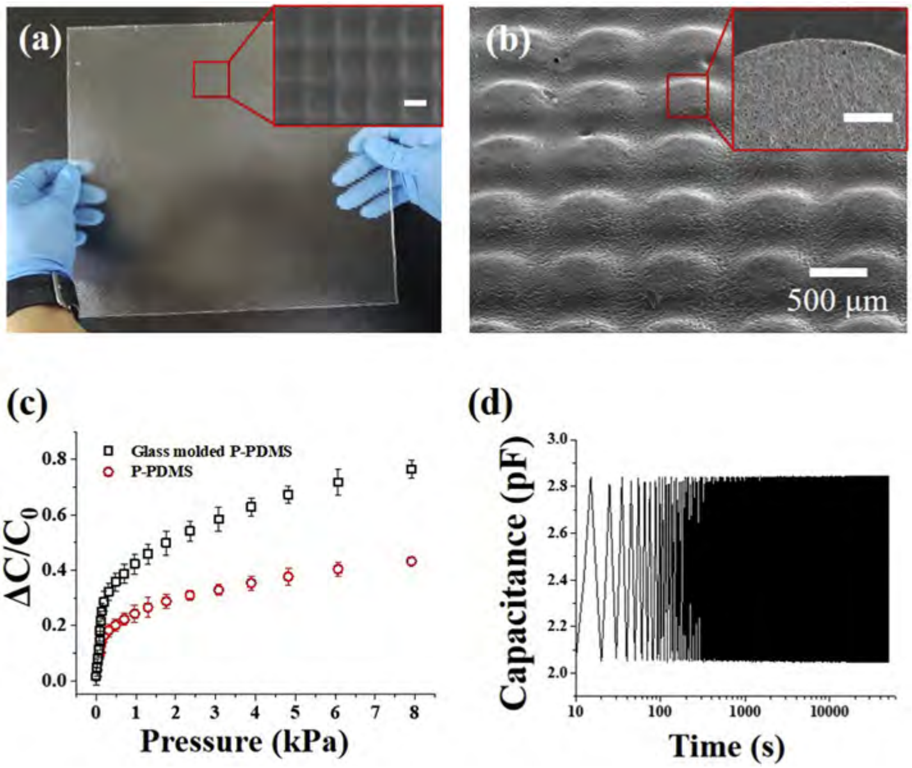

Fig. 4. (a) A photograph of the opaque glass mold. The inset image is the SEM image of the surface, and the scale bar is 500 μm. (b) PDMS film with patterns of transferred mold. The inset image is the cross-sectional view of the film, and the scale bar is 200 μm. (c) Capacitance change ratio of the sensor with and without microstructures on the dielectric layer, and error bars indicate standard deviation of three samples. (d) Output signals of the proposed sensor under the pressure of 0.2 kPa during 5000 loading-unloading cycles.

the proposed sensor using a cyclic test. A glass molded P-PDMS dielectric layer with a solvent ratio of 40 wt% was used for the test. The applied pressure was fixed at 0.2 kPa, and 5000 loading-unloading cycles were implemented at 0.1 Hz. As shown in Fig. 4(d), the sensor stably maintained the output signals without any remarkable degradation.

3.4. Application of the pressure sensors

We first investigated the performance of a single sensor fabricated with micro-structured P-PDMS. A wire connector with a mass of 0.118 g was placed on the sensor (Fig. 5(a)). The response time is defined as the time interval of 10% through 90% of the steady-state value [29], and the sensor responded in 70 ms as shown in Fig. 5(b). Based on the measured mass, the pressure was calculated to be 0.0115 kPa, and the capacitance change ratio was 0.0239. Thus, the sensitivity of 0.0115 kPa was estimated to 2.07 kPa^{-1}. The sensitivity value was slightly higher than the experimental result in Fig. 4(c) because the pressure exerted by the object was lower than 0.2 kPa. The sensitivity of the capacitive pressure sensor with an elastomeric material is generally higher in the lower and narrower pressure range, so this result is considered reasonable.

The proposed sensor can be easily extended to a sensor array. It is only necessary to make a film and cut it to a size that covers all the pixels. Fig. 5(c) presents a photograph of a 4 by 4 sensor array with 3 mm × 3 mm sized pixels. We placed an object (0.21 g) on the B1 position of the array and placed another object (1 g) between C3 and D3, slightly offset to D3, and measured the change in capacitance. The pressures exerted by the objects were well-identified as shown in Fig. 5(d). It should be noted that D3 showed a larger capacitance change ratio than C3 because the object was closer to D3. The values of C3 and D3 were also valid considering that the pressure was dispersed in the middle part of C3 and D3. The proposed sensor can be easily implemented in the form of a sensor array and can be used to measure the pressure distribution of a large object such as baseball bats, balls, runner's shoes, and chairs.

We also demonstrated the measurements of the static and dynamic pressures of fingers as a skin-attached sensor. First, as shown in Fig. 6(a), we attached a single sensor onto one finger and consecutively tapped a table surface with the finger. Three sets of tapping were conducted with increasing pressure, and the table was tapped four or five times with similar pressure at each set. The sensor detected each tapping well (Fig. 6(b)). The capacitance values below C_0 were measured due to the sticky nature of the PDMS substrates. Then, we further extended this finger sensor by attaching it to all five fingers and picking up an object (Figs. 6(c) and (d)). A 10.55 g plastic cup was held with enough pressure to prevent it from falling, and the capacitance change ratio was measured at each finger (Fig. 6(e)). The capacitance change ratio was the highest at the second finger sensor, and the fifth finger showed the lowest value. This means that the experimenter gave the index finger the greatest force and the little finger the smallest force while holding the cup. From this demonstration, it would be expected that the proposed pressure sensor could be applied to artificial skin or medical instruments.

4. Conclusion

In summary, we proposed a flexible, highly sensitive capacitive pressure sensors using 1) P-PDMS dielectric layers fabricated by simple microwave treatment and 2) surface microstructures transferred from very low-cost, commercial opaque glass plate. The sacrificial solvent ratio was directly related to the pore size and porosity, which in turn strongly influenced the sensitivity of the pressure sensor. The sensitivity as high as 0.813 kPa^{-1} was obtained at the solvent ratio of 40 wt% due to larger deformation caused by lower Young's modulus of the P-PDMS layer. With the help of microstructure formed on the surface of P-PDMS, the sensor had further enhanced the sensitivity of 1.43 kPa^{-1} and a fast response time of 70 ms under a small object as light as 0.118 g. The performance of a sensor array was measured by placing objects at different locations, and the finger sensors were also demonstrated by tapping a table and grabbing a plastic cup. The proposed pressure sensor using both simple microwave treatment and very cheap surface

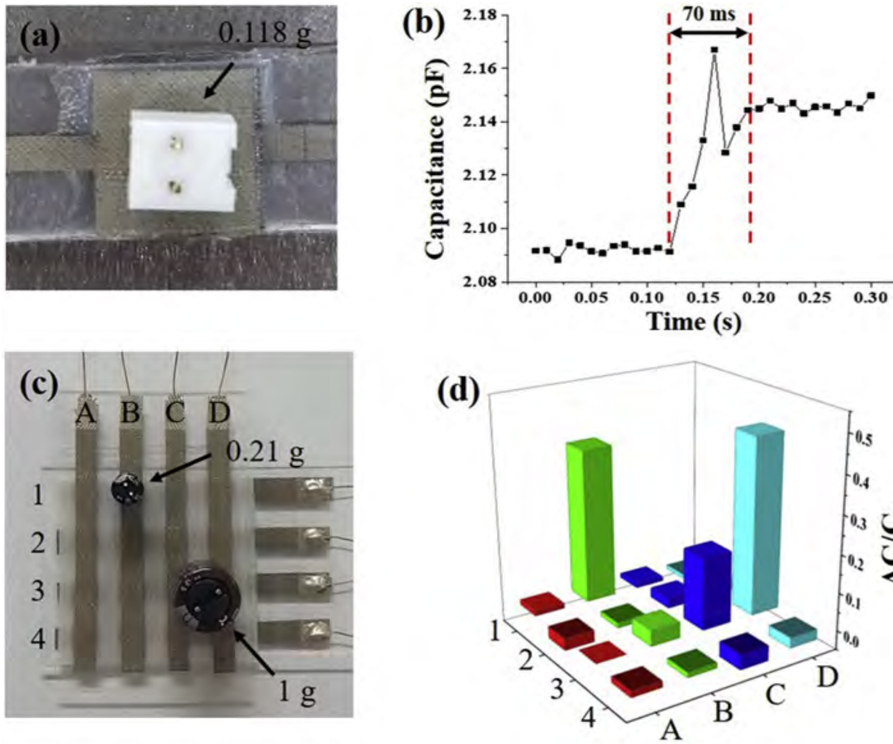

Fig. 5. Pressure sensor with a micro-structured dielectric layer and pressure sensing. (a) A photograph of pressure loading and (b) response time of the sensor. (c, d) 0.21 g and 1 g weights were placed on the sensor array, and the resulting capacitance change ratio is shown.

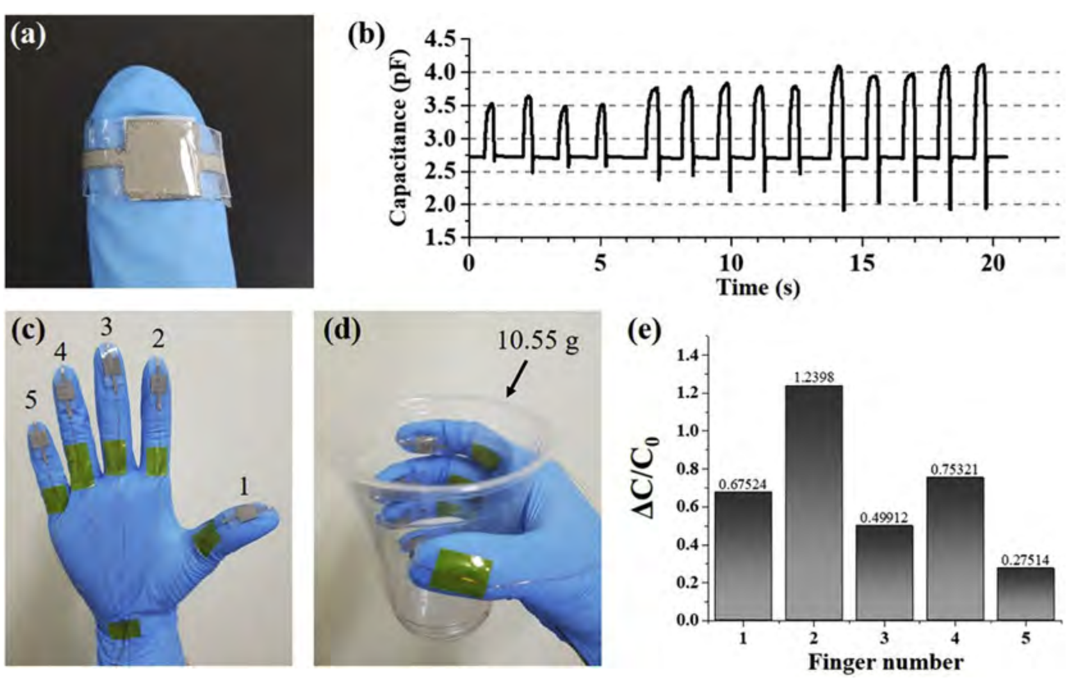

Fig. 6. (a) A photograph of a sensor attached to a finger and (b) capacitance values in gentle tapping. (c, d) Five sensors on each finger for grabbing a plastic cup and (e) capacitance change ratio.

microstructure could be easily extended to large-area sensor arrays or skin-attachable sensors, and hence utilized in the application of soft, flexible and stretchable electronics.

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