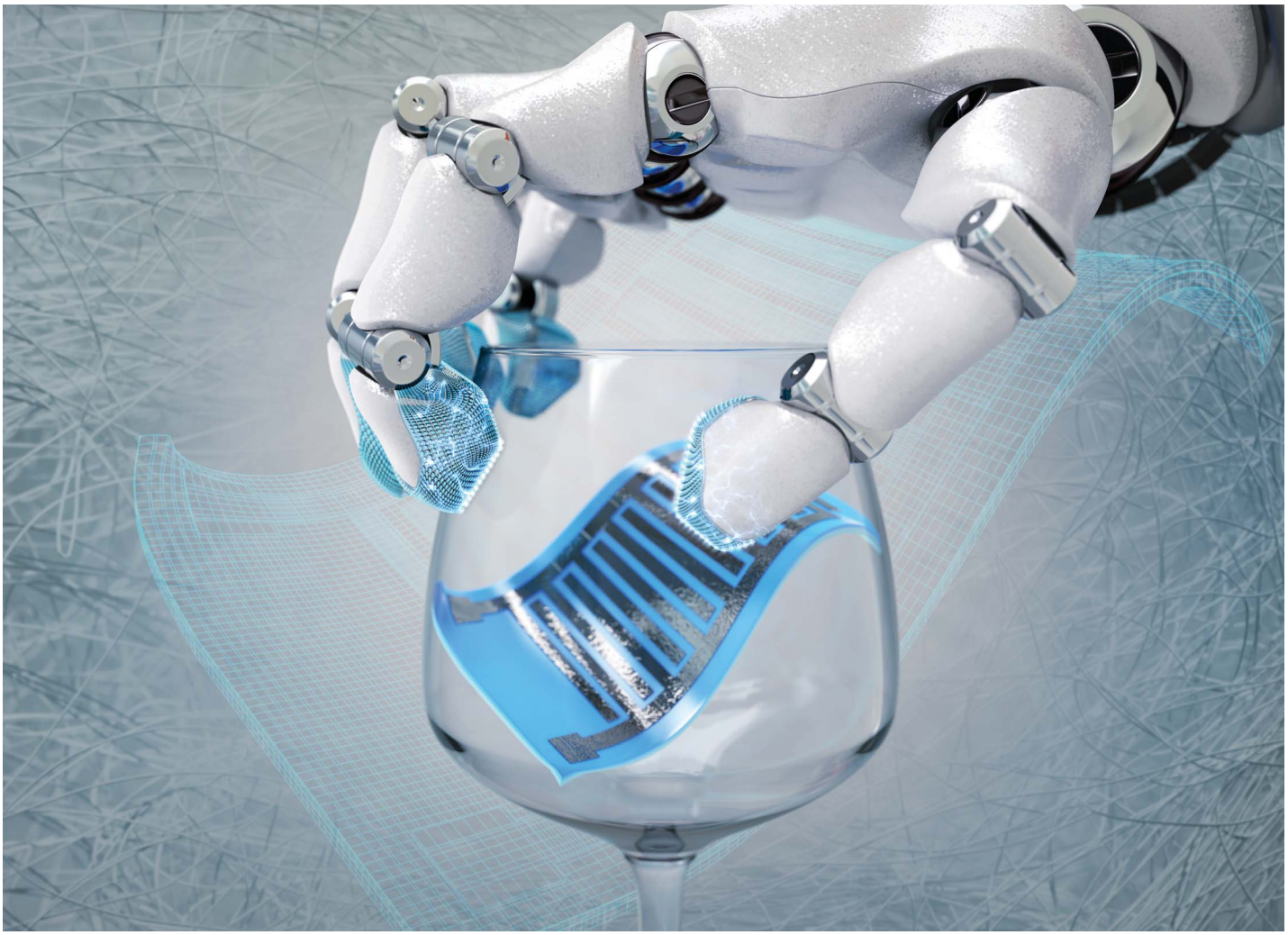

Showcasing research by Dr Jong-Woong Kim at Korea Electronics Technology Institute, Republic of Korea.

A wearable piezocapacitive pressure sensor with a single layer of silver nanowire-based elastomeric composite electrodes

A transparent, stretchable and wearable pressure-sensitive capacitor was developed by employing a single layer of Ag nanowire-based elastomeric electrode.

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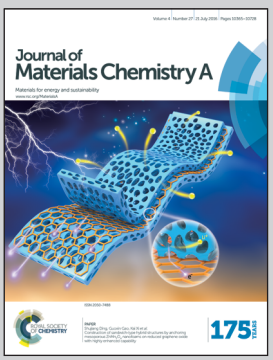

See Byeong-Kwon Ju, Jong-Woong Kim *et al.*, *J. Mater. Chem. A*, 2016, **4**, 10435.

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A wearable piezocapacitive pressure sensor with a single layer of silver nanowire-based elastomeric composite electrodes†

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A highly stretchable and transparent pressure sensor based on a single layer silver nanowire (AgNW) electrode was successfully fabricated. In order to achieve this, we synthesized a stretchable and transparent adhesive, polyurethane urea (PUU), which was designed to firmly adhere to the AgNWs by incorporating 2,2-bis(hydroxymethyl)butyric acid with a carboxylic group. A patterned AgNW-based sensing electrode could be completely transferred to a PUU film deposited on a poly(dimethylsiloxane) (PDMS) substrate, resulting in a AgNW/PUU/PDMS pressure-sensitive capacitor. The capacitance was developed on a AgNW tandem compound electrode pattern by the fringing effect, which decreased with increasing pressure applied to the surface of the sensor. The sensitivity did not significantly deteriorate, even when the sensor was stretched up to a strain of 35% or when the stretching was cycled up to 10 000 times. A wearable acupressure sensor with a pressure sensitive matrix of nine pixels was successfully demonstrated.

Introduction

Recently, the research society has developed wearable devices that can quantitatively measure an external pressure in response to physical contact.[1–8] Such technology could be used in future applications such as robotic systems, electronic skin, prosthetics, and wearable medical devices.[9] Advanced sensors can help robotic and prosthetic devices "learn" how objects feel during interactions and obtain biosignals from finger touches or body motions used to control the robotic or prosthetic devices.[10] In order to achieve wearable pressure-sensitive devices, the pressure sensing mechanism should first be defined and designed to establish an appropriate gauge factor, and then the corresponding sensor design and suitable materials can be selected. To date, various methods have been proposed to achieve pressure sensitivity based on piezoelectric,[11–13] piezoresistive,[14,15] triboelectric,[16,17] and piezocapacitive materials.[18–21] The majority of these techniques use the changes in electrical parameters (such as conductivity and impedance) or geometry of a specific material produced by small mechanical deformations. The gauge factor is defined as the ratio of the relative changes in the measured property to the mechanical strain, and a high gauge factor (*i.e.*, sensitivity) is desirable.

Piezocapacitive sensors transduce a contact with a conductive object (such as a human finger) into a change in capacitance, and they have been very widely used in conventional touch sensors as they have a simple read-out mechanism.[22] Capacitive sensors are based on capacitive coupling with a conductive object. This mechanism is very sensitive and even extremely weak interactions with other objects can be detected. Recently, piezocapacitive materials have been used to fabricate pressure sensors.[18–21] A popular device design uses a layer of a soft dielectric polymer sandwiched between two flexible transparent electrode layers, where the capacitance is formed between the electrodes. When a pressure is applied to the sensor, the distance between the two electrodes is reduced, resulting in an increase in the capacitance, which can be used as a sensing parameter. This design requires the fabrication of patterned transparent electrode layers, which are fixed to the polymer using a transparent adhesive, resulting in a fully transparent pressure sensor that could be used in fabrications of pressure sensitive flexible displays as well as wearable biocompatible sensors. Considering that the flexibility of the components (ability for the material to be stretched) often determines wearability of devices, all of the materials, including the electrode and the adhesive, should be highly flexible. However, the poor processability of stretchable polymers such as poly(dimethylsiloxane) (PDMS), polyurethane, and polyurethane acrylate originating from their low surface energies prevents the practical use of these materials for this application.[23] This implies that a simple and more reliable structure with an affordable fabrication method needs to be developed. Furthermore, the poor adhesion between the polymers and

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nano-sized conductive fillers, such as carbon nanotubes, graphene, and silver nanowires (AgNWs) needs to be resolved as it makes it difficult to achieve stable flexibility.

To address these current limitations in the development of stretchable piezocapacitive pressure sensors, here we propose a novel fabrication method using a single layer AgNW-based elastomeric electrode. A simple tandem compound electrode was patterned on AgNW electrodes, allowing a capacitance to be formed between the parallel electrodes, which varies with the gap. To attain a mechanically stable, reliable, transparent, and stretchable sensor, a flexible transparent adhesive was synthesized to enhance the adhesion between the AgNWs and the polymer. Finally, we tested the feasibility of this new approach by fabricating simple, stretchable, and transparent pressure sensors that could be used to weigh various objects and quantify the forces applied by a fingertip.

Experimental section

Materials

Polyester diol (number-average molecular mass, $M_w \approx 1000$) was purchased from Songwon, Korea. 2,2-Bis(hydroxymethyl) butyric acid, isophorone diisocyanate, dibutylamine, toluene, and 2-propanol were purchased from Sigma-Aldrich Chemicals, USA. Isophorone diamine was purchased from Tokyo Chemical Industry, Japan. A SYLGARD 184 elastomer kit was purchased from Dow Corning, USA. All chemicals were used as received without purification.

Synthesis

The synthesis scheme for the transparent and stretchable adhesive (polyurethane urea; PUU) is summarized in Fig. S1.† A mixture of polyester diol (82.8 g, 82.8 mmol), 2,2-bis(hydroxymethyl)butyric acid (1.6 g, 10.8 mmol), and isophorone diisocyanate (29.0 g, 130.4 mmol) in toluene (82.8 g) was heated at 90 °C for 3 h. The solution was then cooled to room temperature (RT), toluene (60 g) was added, and the viscous solution was heated to 70 °C. To this solution was added a mixture of isophorone diamine (5.4 g, 31.7 mmol), dibutylamine (0.6 g, 4.6 mmol), toluene (115.2 g), and 2-propanol (68.4 g). The solution was stirred at this temperature for 3 h and cooled to RT. Further addition of a mixture of toluene (28.8 g) and 2-propanol (14.4 g) resulted in a solution with a solid content of 25%. The synthesis method and analysis of the product are described in more detail in our previous study.[24]

Pressure sensor fabrication

The fabrication of the AgNW/PUU/PDMS-based transparent and stretchable sensor is illustrated in Fig. 1. A polyimide film (Kapton, DuPont, USA) was first cleaned using detergent, deionized water, and isopropanol. A solution of AgNWs dispersed in isopropanol (Nanopyxis Ltd., Korea) was spin-coated onto the Kapton film and heated on a hot plate at 60 °C for 10 min to remove any remaining organic solvent from the coated layer. Before patterning the AgNWs, an intense-pulsed-light (IPL, 1.8 kV input voltage) was used to irradiate the AgNW electrodes for a pulse duration of 300 μs using a photonic sintering system (Sinteron 2000, Polytec Ltd., USA). This process was shown to slightly enhance the adhesion between the AgNWs and the Kapton. To induce capacitive coupling on a single AgNW electrode layer, a tandem compound electrode pattern was designed, as shown in Fig. S2.† The tandem compound pattern was prepared using photolithography, after which the AgNWs were etched with an acidic solution (chromium etchant, Sigma-Aldrich, USA), followed by stripping of the photoresist (AZ1512, Merck, Germany). Point-to-point

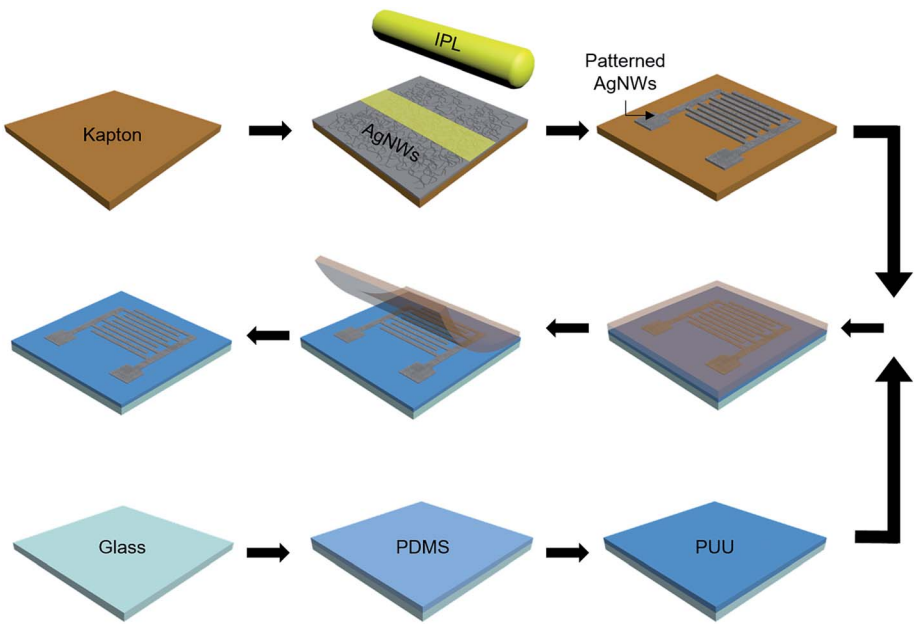

Fig. 1 Schematic of the fabrication procedure for AgNW/PUU/PDMS-based pressure sensors.

resistance measurements revealed that the patterning process did not significantly degrade the electrical performance (≤10% drop in conductivity).

In a separate procedure, 200 μm PDMS films were fabricated using the elastomer kit (w/w = 1 : 10). Oxygen plasma treatment was employed to form hydroxyl functional groups on the PDMS film. O_2 gas flow rate, gas pressure, power and treating time were controlled to be 30 ml min^{-1}, 15 Pa, 50 W and 120 s, respectively. The synthesized PUU was then coated on the PDMS using spin coating, followed by drying of the solvent, resulting in a thickness of 8 μm. The patterned AgNW electrodes on the Kapton films were adhered to the PUU/PDMS layers with the patterned side down using a roll laminator heated to 80 °C. After cooling, the AgNW/PUU/PDMS film was peeled off the Kapton film. After the transfer procedure, three cycles of IPL irradiation with a higher voltage (2.0 kV) and a longer pulse duration (500 μs) were applied to the AgNW/PUU/PDMS samples, in order to further enhance the adhesion between the nanowires and PUU.

Characterization of the sensor

A field-emission scanning electron microscope (FESEM; JSM6700F, JEOL Ltd., Japan) was used to investigate the microstructure of the AgNW networks. Optical transmission was also measured using a UV-visible spectrophotometer (V-560, Jasco, Japan), while the sheet resistance (R_s) was measured using a non-contact measurement system (EC-80P, Napson Corporation, Japan). The surface morphology was measured using atomic force microscopy (AFM; XE-100 TM, Park Systems, USA). An automatic stretch-testing machine (Jaeil Optical System, Korea) was used to measure the long-term reliability under repeated stretching cycles. The electrodes were elongated at a rate of 5 mm s^{-1}, and their resistance was measured during both stretching and contraction. An inductance–capacitance–resistance (LCR) meter (HP4284A, Agilent, USA) equipped with a probe station (MST8000C, MSTech, USA) was used to measure the capacitance of the static samples. The dynamic capacitance of the sensors was measured using an oscilloscope (ZM2353, NF, Japan). Pressure sensing tests were conducted with an equipment fabricated in house. The pressure was applied either directly onto the sample or through various cover layers (assuming the sensor needs to have a protective layer). We employed various dielectric materials, such as a 125 μm thick polyethylene terephthalate (PET) film, a 100 μm thick glass plate, and a 300 μm thick glass plate, to cover the sensor for these tests. More than 10 samples were fabricated and measured for determining most parameters.

Results and discussion

PDMS is considered an excellent candidate material for flexible substrates of various electronic and bio-sensors, because of its high transparency, neutral color, high elasticity, large elongation up to 160–180%, and biocompatibility.[25,26] Despite such advantages, its low surface energy (and hence, poor adhesion to other materials) makes its use as a substrate for stretchable electrodes challenging. For instance, when a AgNW network is formed on PDMS, it delaminates easily from the polymer with gentle rubbing or moderate mechanical stress. To successfully deposit AgNWs onto the silicone film, the surface of PDMS was treated with an oxygen plasma to enhance its wetting properties (increase the surface energy). After this surface treatment, the AgNW dispersion could be more uniformly deposited on the polymer, but the adhesion remained poor. The PUU film synthesized here was designed to adhere well to both the AgNWs and PDMS. Adding 2,2-bis(hydroxymethyl)butyric acid to PUU was expected to enhance the affinity of AgNWs for PUU, in which the carbonyl groups of poly(vinylpyrrolidone) (PVP) at the surface of AgNWs presumably formed hydrogen bonds with the carboxylic acid groups of PUU. The surface of PDMS, which became hydrophilic after the plasma treatment, was coated with PUU, where hydrogen bonding between the hydroxyl groups and urea (or urethane) was expected to enhance the interlayer interaction. By employing PUU as an adhesion layer, the AgNWs could be very stably adhered to PUU on PDMS, resulting in a flexible transparent electrode.

As shown in Fig. 1, we first deposited the AgNWs onto a preliminary Kapton substrate to form a uniform AgNW network. The poor adhesion between the AgNWs and Kapton is well known and facilitated the easy transfer of the nanowire layer to the target substrate (PUU coated PDMS).[27,28] However, the poor adhesion hinders photolithographic patterning and subsequent metal etching because the AgNW layer could be easily removed from the polymer during the development or removal of the photoresist after patterning. Considering that the design proposed in this study, namely a tandem compound pattern as shown in Fig. S2,† consists of fine line widths and gaps of 100 μm, the low adhesion was a major problem. In an attempt to resolve this issue, the AgNW/Kapton film was treated with IPL to enhance the adhesion as demonstrated in a previous study. However, we observed that when the irradiation power was too low (below 1.5 kV), the adhesion of the AgNW film was not sufficiently enhanced for successful lithography. Furthermore, when the irradiation energy was too high (above 2.2 kV), the nanowires were not completely transferred to PUU/PDMS. After extensive consideration of these factors and experimentation, the process parameters were optimized: power input of 1.8 kV, pulse duration of 300 μs, and a single irradiation treatment. By employing these conditions, the AgNW on the Kapton could be more strictly patterned as shown in Fig. S3.† The patterning resolution is also summarized in Fig. S4.†

The AgNW/Kapton films were face-to-face laminated with the PUU/PDMS substrates prepared separately using a roll laminator. The AgNW films were perfectly transferred from the Kapton to the PUU surface, leaving no observable AgNW residues on the Kapton. A comparison of point-to-point resistance values of the electrodes before and after transference revealed that the conductivity of the patterned electrode was not degraded by this process.

The sintering process with high energy pulsed light applied to the AgNW electrodes induced plasmonic heating, and thereby increased the temperature of the underlying polymer as well as the AgNWs.[29–31] This resulted in the local fusion of the

polymer underneath the metallic wires and enhanced the adhesion between the AgNWs and polymer as shown in Fig. S5.† Fig. 2a shows the SEM micrograph of the AgNW/PUU/PDMS surface with the nanowires embedded into the underlying PUU. The nanowires conformed well to the rough surface of PUU, implying that the AgNW film was in good contact with PUU during the lamination step. The relatively high roughness values shown in Fig. 2b were a result of severe local strains induced by the delamination forces at the end of the lamination, forming many ripples on the surface of the electrode. As shown in Fig. 2c, a macroscopic view indicates that the pattern edges were very sharp, and neither defects nor a gradient in AgNW density was observed. In terms of nanowire density, we used a dilute AgNW dispersion of 0.8 wt%, resulting in an R_s value of about 8 Ω sq.$^{-1}$ and a transmittance of about 79% (Fig. 2d). These results are much better than those of commercial indium tin oxide (ITO) films, which are also not flexible or stretchable.

Our device was designed to form capacitors using the parallel line electrodes. We first measured the capacitance value without applying any pressure. A capacitance of about 12.2 pF was measured by the sensor, originating from the electromagnetic coupling of the parallel electrode lines. This value is somewhat smaller than that typically formed on a commercial capacitive touch sensor consisting of two ITO layers (about 30–50 pF). In our case, there is no direct capacitance formed between the adjacent electrodes (only fringing capacitance is present), because of the absence of confronting areal electrodes in our single layer sensor. We applied compressive pressure to the surface of the sensor with an object (an 8 mm diameter aluminum cylinder coated with an insulation layer) to investigate the variation in capacitance with applied pressure. Fig. 3 shows the change in capacitance measured as a function of applied pressure, up to 230 kPa, with different dielectric cover layers (see also Fig. S6† for capacitance change with smaller pressure).

As can be seen in Fig. 3, the capacitance decreased with increasing pressure with high sensitivity regardless of the covering material. Not surprisingly, the rate of decrease (slope of the curve) is highest in the case of direct pressure (no cover layer) followed by the thin PET layer, thin glass, then thick glass. This finding demonstrates that the decrease in capacitance is dominated by the mechanical deformation of the sensor. Using the 125 μm thick PET formed a larger gap between the sensing electrode and the contact object than the thin glass (100 μm), but the sensitivity was higher for the polymer films. This implies that pressure applied through a soft dielectric material can be more effectively sensed than through a hard material, presumably due to the larger local deformation. Covering the sensor with a much thicker glass (300 μm) likely distributed the

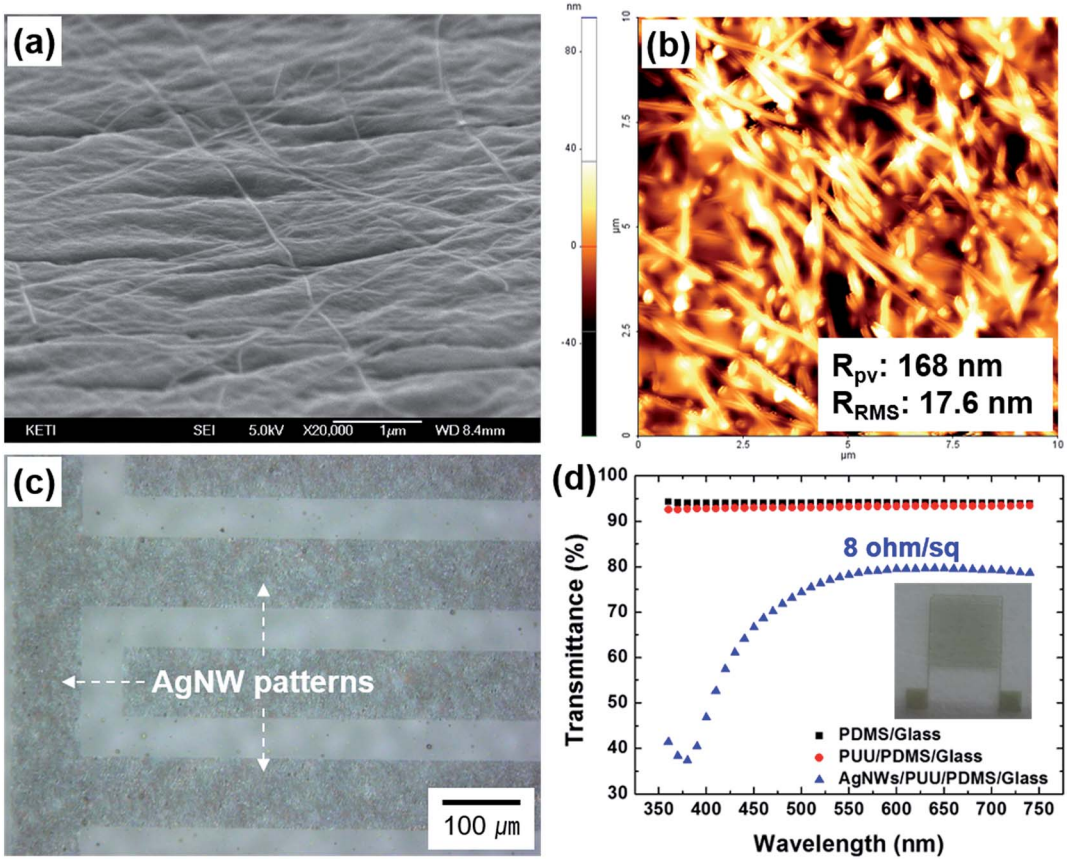

Fig. 2 (a) SEM and (b) AFM micrographs of the AgNWs transferred onto the surface of PUU/PDMS substrates, (c) AgNW electrode patterns, and (d) measured transmittance of PDMS/glass, PUU/PDMS/glass, and AgNW/PUU/PDMS/glass. The inset is a photograph of the fabricated pressure sensor.

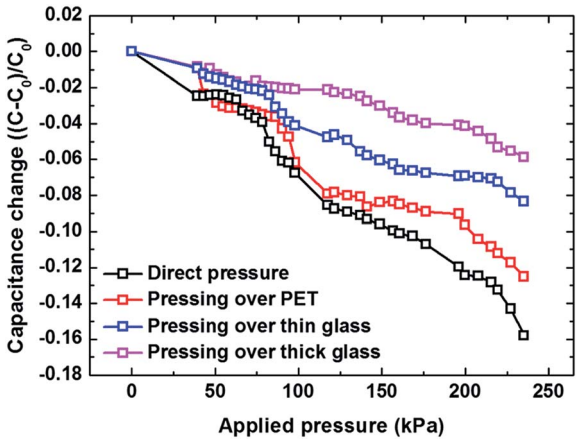

Fig. 3 Change in the capacitance of the fabricated sensor as a function of applied pressure up to 230 kPa, using various cover materials.

stresses more widely, so that the localized deformation and hence, sensitivity decreased. However, even for this worst case, the capacitance decreased about 6% with the application of 230 kPa pressure, which is thought to be sufficient sensitivity for such a sensor.

A suggested mechanism for the decrease in capacitance with applied pressure is schematically illustrated in Fig. 4. Before the object touches the sensor, the parallel electrodes are electromagnetically coupled, mainly producing a fringing capacitance (Fig. 4a). Upon touching the surface of the sensor, the object is instantly coupled with the electrodes, resulting in a decrease in the existing capacitance (Fig. 4b). A mutual capacitive sensor commercially available in the market uses this drop in capacitance for sensing finger touches. By applying a slight pressure to the sensor, the surface of the sensor locally deforms, increasing the gap between the parallel electrodes (Fig. 4c). This gap increases with higher applied pressure as illustrated in Fig. 4d. Considering that the capacitance is dominated by the fringing effect (which is inversely proportional to the gap between the electrodes), the capacitance drop with increasing pressure is reasonable. This could be observed in Fig. S7,† in that the capacitance was more largely varied by pressing with a smaller object (severer mechanical deformation of the sensor could be induced by pressing with tiny objects). This mechanism also explains the effect of the covering materials on the capacitance, shown in Fig. 3. Less flexible cover materials will result in smaller changes in the capacitance as the local deformation is smaller due to a well distributed stress. In other words, more flexible (softer and thinner) cover layer materials allow higher deformations and larger drops in capacitance. Considering the substrate, the material needs to be sufficiently soft and thick to be easily deformed.

We tested the reproducibility of the sensing properties by applying and increasing pressure up to 250 kPa and then releasing the load. Fig. 5a shows the capacitance change measured during these tests. The capacitance decreased with increasing pressure, and recovered to its original value after the load was released. Only slight hysteresis was observed, which means that the fabricated sensors behave nearly elastically. Cycling the load up to five times showed reproducible behavior, as plotted in Fig. 5b. The capacitance consistently decreased with an increase in applied pressure, and similar values were reproduced up to 1000 cycles, demonstrating the high reliability and reproducibility of the fabricated sensor.

Given that the primary goal of this study was to achieve highly reliable sensing performance under conditions of

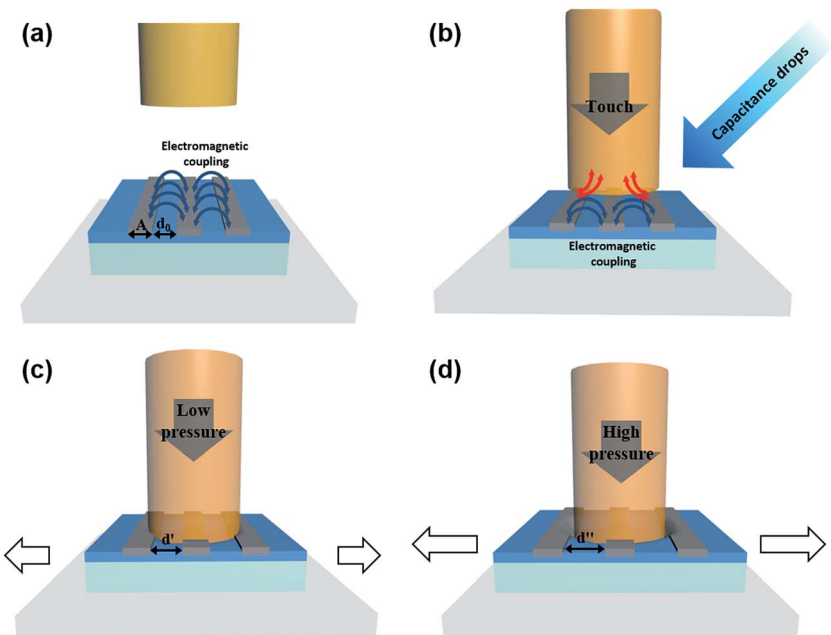

Fig. 4 Suggested mechanism for the varying capacitance of the sensor with applied pressure. (a) Before the object touches the sensor, (b) the instant the object touches the surface of the sensor, (c) slight pressure applied by the object, and (d) a higher pressure applied by the object.

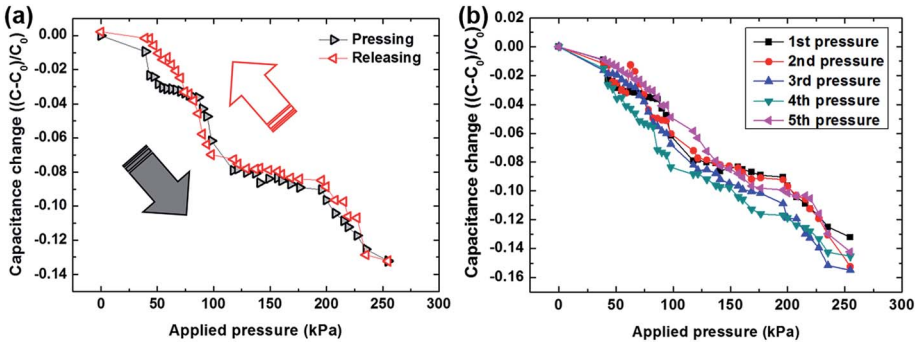

Fig. 5 Capacitance changes of the fabricated sensor measured during compression up to 250 kPa. (a) One pressing and release cycle. (b) Data for five different pressing cycles. In both cases, a thin PET film was used to cover the sensor.

continuous mechanical deformation, various stretching tests were carried out on all of the sensors fabricated in this study, as shown in Fig. 6. Fig. 6a shows the electrode design with the definition of the *x* and *y* axes for the stretching measurements. First, we investigated the effect of applied strain on the capacitance of the sensor. As shown in Fig. 6b, stretching in the *x* direction resulted in increased capacitance due to the reduced gap between the parallel electrodes. In contrast, stretching in the *y* direction increased the gap, resulting in a decrease in the capacitance. Stable capacitance values were observed even when the samples were stretched by 35%, varying only 9% and 12% compared to the unstretched state when stretched in the *x* and *y* directions, respectively. In addition, even after the samples were repeatedly stretched (30% strain in the *y* direction) up to 1000 times, the capacitance decreased only 2%, as shown in Fig. 6c. After 10 000 stretching cycles, a decrease in the capacitance around 10% was observed but the pressure sensing capability still performed well. This implies that the fabricated sensor has excellent flexibility and mechanical stability, which is attributed to the good adhesion between the AgNW and PUU films as a result of the functionalization of the polymer and the increased interfacial area produced by IPL irradiation.

Fig. 7a shows the capacitance changes induced by various interactions with a gloved human fingertip while the sensors were stretched in the *y* direction (as shown in Fig. 7b and c). Interestingly, nearly the same relative capacitance drop was measured for all samples regardless of the applied strain, which means that the ability of the sensor to detect the applied pressure did not deteriorate when stretched. To the best of our knowledge, this is the first example of the pressure sensor demonstrating high sensitivity in a stretched state, in that the sensor was fabricated using a single layer of AgNWs well adhered to the stretchable and transparent adhesive on PDMS.

Conventional piezoresistive or piezocapacitive sensors cannot be uniformly adhered to a 3-dimensionally curved object such as a fingertip or contact lens due to their poor flexibility. The sensor fabricated here could be attached to a human fingertip without forming any wrinkles, thanks to the high stretchability. In addition, the good performance under highly stretched conditions could allow our sensors to be uniformly wrapped around an object, facilitating various new applications. For example, a sensor attached to a human fingertip could be used as an acupressure sensing system.

Fig. 8a–d show an example where the sensor is being used on a gloved finger, demonstrating that the measured capacitance decreases with the increase in pressure required to compress a balloon. Considering that there may have been many sources of interference (noise) from circuits or interconnects, as we did not employ any shielding apparatuses to block the parasitic electromagnetic coupling with the environment, the sensor functioned very reliably, giving reproducible data. When the fingertip contacted the surface of the balloon, the capacitance decreased by about 5%, and then further decreased with increasing pressure. This demonstration shows the high potential of our sensor system for developing various biocompatible wearable sensors. Another demonstration in Fig. 8e

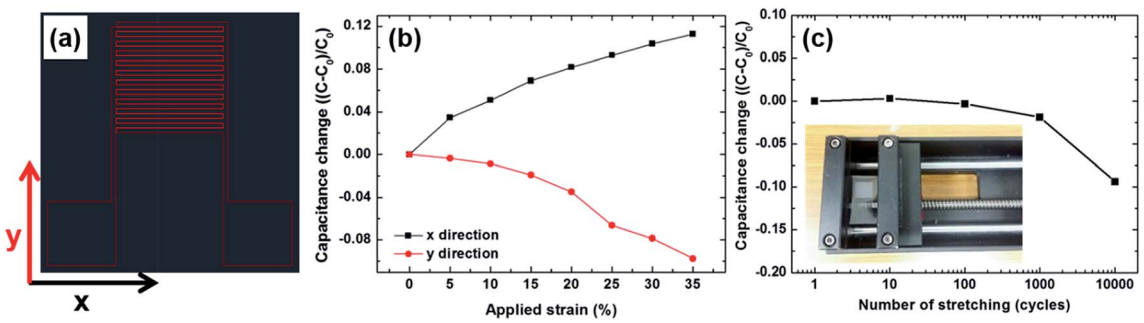

Fig. 6 (a) A capacitor design indicating the *x* and *y* axes for the stretching tests. (b) The effect of strain applied in the *x* or *y* direction on the capacitance change. (c) The effect of repeated stretching in the *y* direction on the capacitance change, up to 10 000 cycles.

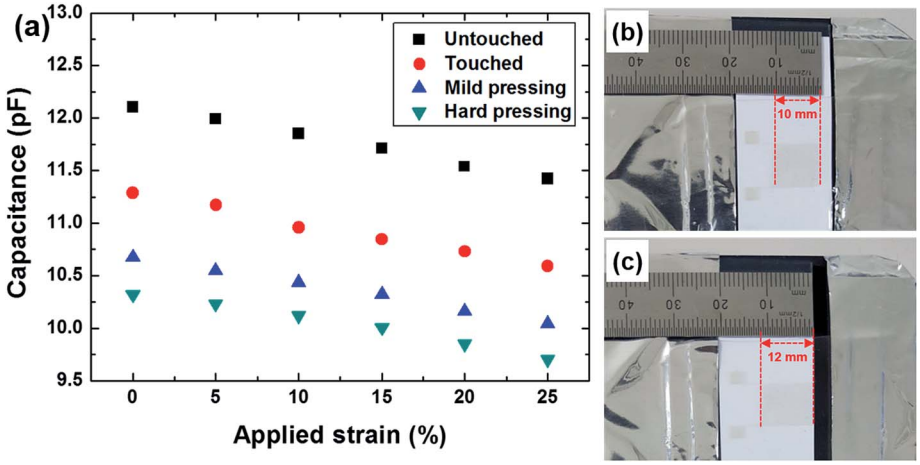

Fig. 7 (a) Capacitance of the sensors stretched in the *y* direction under conditions of "untouched", "touched", "mild pressing", and "hard pressing" by a gloved human finger. (b) and (c) show the fabricated sensor before and after stretching, respectively.

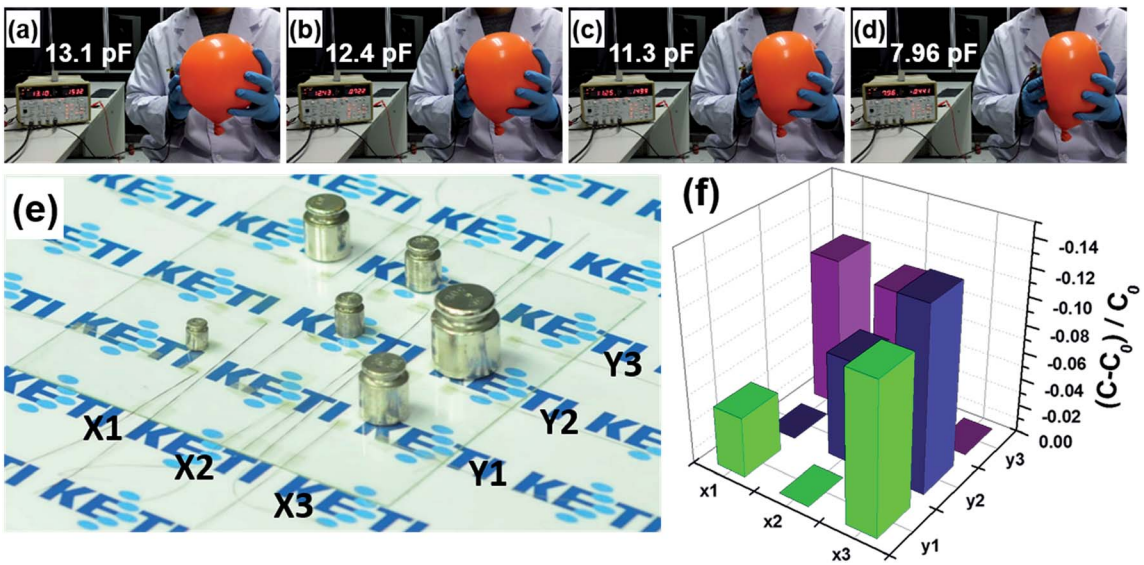

Fig. 8 (a–d) Measured capacitance of the sensor attached to a fingertip while applying pressure to a balloon. (e) 3 × 3 sensor matrix with six different weights placed on the pixels. (f) Absolute values of the capacitance as a function of the location of the weights.

shows a pressure sensitive matrix of nine pixels with a pixel dimension of 10 mm by 10 mm and a pitch of 30 mm. The sensor array monitored the weight distribution on each pixel by detecting the change in capacitance. Fig. 8f shows a capacitance map, where the position and weight on each pixel were identified by measuring the capacitance values. If we consider that the capacitive sensor is very sensitive to parasitic capacitances, it was needed to observe the parasitic effects on the sensing capability of our sensor. As could be seen from Fig. S8,† the capacitance change was not largely affected by the application of pressure onto a neighboring pixel, mainly due to the large pitch of the pixel design (30 mm). Fig. S9† indicates that the capacitance change was being affected by decreasing the distance between pressing objects to smaller than 10 mm. If the pressure applied to the adjacent pixel was increased to make the mechanical coupling, the mechanical crosstalk should be larger and the influential distance could be increased.

With these examples, we have demonstrated that we did not need to employ complicated or expensive procedures to enhance the adhesion of the nanowires to the stretchable polymer. Nor was it necessary to sandwich a dielectric material between two separate electrodes to realize a mechanically stable, stretchable, and transparent sensor. We verified that only a single layer of patterned AgNW electrodes, well attached to a stretchable polymer substrate, was all that needed to achieve a high performance pressure sensor.

Conclusion

A highly stretchable and transparent pressure sensor was fabricated using a single layer of patterned AgNWs, a flexible and transparent adhesive (PUU), and a freestanding polymer (PDMS). AgNW electrodes patterned on a preliminary substrate (Kapton) were completely transferred to the target substrate

(PDMS) due to the good adhesion with PUU. The incorporation of 2,2-bis(hydroxymethyl)butyric acid with a carboxylic group into PUU gave the high adhesion between the AgNWs and PUU. In addition, PUU adhered strongly to PDMS, where hydrogen bonding between the hydroxyl groups and urea (or urethane) enhanced the interlayer interaction. This resulted in a mechanically stable and stretchable electrode with a transmittance of 79% and R_s value of 8 Ω sq.$^{-1}$. By designing a simple tandem compound pattern, a capacitance was formed by the fringing effect, which decreased with increasing pressure applied to the surface of the sensor. The pressure sensitivity of the sensor was reproducible, even when it was stretched up to 35%. To the best of our knowledge, this is the first report of a capacitive pressure sensor demonstrating sensitivity under such large mechanical deformations. By virtue of this, we have shown two interesting demonstrations of potential applications; the sensor could weigh various objects placed on its surface or measure the forces applied by a fingertip to an object. Considering that the sensor fabrication method is simple and easily scalable, we believe that it can be applied to a variety of stretchable and wearable devices.

Acknowledgements

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