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LETTER

Wearable piezoresistive strain sensor based on graphene-coated three-dimensional micro-porous PDMS sponge

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Abstract

We present a highly elastic and wearable piezoresistive strain sensor based on three-dimensional, micro-porous graphene-coated polydimethylsiloxane (PDMS) sponge suitable for being attached on human skin. The proposed strain sensors are simply fabricated by a sugar templating process and dip coating method based graphene ink in a facile and cost effective manner. The fabricated graphene-coated PDMS sponge shows highly stable mechanical properties in various tensile stress–strain test. A graphene thin film coated onto the backbone of PDMS sponges is used as the sensing materials of piezoresisitve strain sensors. The changes in resistance of the devices are highly stable, repeatable, and reversible when various strain is applied. Furthermore, the strain sensors show excellent sensing performance under different strain rate and mechanically robustness enough to be worked stably under repeated loads without any degradation.

Keywords: Graphene, PDMS, Strain sensor, Porous sponge

Introduction

Recently, there has been attracting attention in order to develop wearable sensors that can be applied to variety of applications including electronic skin [1, 2], soft robotics [3–5], health monitoring [6, 7], and human–machine interfaces [8]. For these fields of wearable applications, it is greatly important that to make various key factors such as low fabrication cost, sensitivity, long-term stability, and highly conformable properties with high performance. Conventionally, strain gauge based on metal foil have been widely used to measure deformation or strain on such objects. Even though fabrication process of conventional strain gauges is well-established technology such as traditional micro electro mechanical systems (MEMS), they have poor stretchability because they use rigid materials [9, 10]. Due to the rigidity and small

working range, the strain gauges are unable to detect large strain in wearable applications.

To overcome these fundamental limitations of metal strain gauges, research efforts have focused on widening the detection range of strain sensors. The proposed method for the fabrication of sensors is to use of nanomaterials such as Carbon Nanotubes (CNTs), Carbon Blacks (CBs), Graphene, Zinc Oxide Nanowires (ZnO NWs), Siliver Nanowires (AgNWs) that can be coupled with elastic polymer. These nanomaterials based strain sensors is a promising materials of choice due to their excellent mechanical and electrical properties [11–14]. In particular, piezoresistive strain sensors can be manufactured with simple method using elastic polymer with conducting nanomaterials [3, 14–18]. Amjadi et al. [11] suggested a strain sensor with tunable gauge factor (GF) in the range of 2 to 14 based on connection–disconnection of AgNW network in elastic polymer. Lee et al. [19] fabricated a wearable strain sensors for human motion detection based on silver nanoparticle (Ag NP) thin film to change the electrical resistance caused by the opening/

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closure of micro-cracks. Fan et al. [20] reported a highly stretchable strain sensors based on CNTs layer on a thermoplastic polyurethane (TPU) multifilament, especially the strain to failure of 1500%. As such, studies using nanomaterials with elastic polymer, which transduce applied strain into an electrical resistance, have been widely used due to the ease of signal processing. However, the sensors manufactured in the form of thin film suffered from low GF under 10, high hysteresis due to the viscoelastic properties of elastic polymer and slip-page of nanomaterials under stretching/releasing cycles.

Studies on porous sponge have focused on enhancing the sensitivity of the sensors with reduced hysteresis properties. The polymer-based porous sponge, which can be manufactured through various methods such as sugar templating process [14, 21, 22], gas-forming [23, 24], and 3-D printing [25–27], has the advantage of being easily deformed even at a small force and rapidly recovering. The porous sponge undergoes reversible mechanical deformation due to the reduced viscoelastic properties of polymer and anti-barreling phenomenon [14]. Many studies have been proposed to coat or synthesize nanomaterials on porous sponges and use them as sensing materials of strain sensors based on piezoresistivity. Samad et al. [28] proposed a graphene foam with excellent conductivity, reaching 160 S m^{-1} and high strain-sensing sensor using dip coating and pyrolysis. Li et al. [29] also suggested highly flexible and three-dimensional graphene foam, which is capable of 30% of its original length, via self-assembly and dry freezing. However, manufacturing of the three-dimensional graphene sponges for strain sensor requires expensive and complicated temperature control such as pyrolysis or low freezing. Despite the high cost fabrication method, there are limitations in stretchability and reliability due to the low bond force between polymer and graphene. Therefore, it is still a great challenge to study to fabricate a strain sensor with easy of fabrication, high sensing performance, as well as excellent reliability during repeated the stretching/releasing cycles.

In this work, we report a highly elastic and wearable strain sensor utilizing three-dimensional, micro-porous graphene-coated polydimethylsiloxane (PDMS) sponges that are capable of measuring strains. The strain sensor based on graphene-coated PDMS sponge, which is highly conductive, flexible, and stretchable, is fabricated by using sugar templating process and dip coating directly onto a bare PDMS sponge without any complicated process. We measured the mechanical properties of porous sponges including tensile stress–strain curves under different strain range and repeated cycles. To verify the sensing performance of the strain sensor, we checked the change in resistance of the sensor under various

conditions. Our sensor shows an excellent performance of gauge factor as 19.2 at 50% strain, high repeatability, and reliability up to 1000 cycles at 20% strain without any signal degradation.

Fabrication of graphene-coated PDMS sponge

Figure 1 shows the fabrication process of the graphene-coated PDMS sponge using sugar templating process and dip coating. Firstly, sugar and water are mixed in a weight ratio of 30 to 1 and put in a pre-fabricated flexible mold and becomes a stacked sugar. The stacked sugar is heated for 30 min at 70 °C to completely evaporate the water (Fig. 1a) and changes to the cube sugar. A PDMS precursor (Sylgard 184, Dow Corning Corp., USA) with a mixture of resin and hardener in a weight ratio of 10 to 1 is placed in a vacuum chamber (Jeio Tech, Korea) under 1×10^{-1} torr for 10 min to remove the bubbles. The fabricated cube sugar placed onto a Petri dish, and a PDMS was poured into the Petri dish. The PDMS precursor fills the empty space in the cube sugar through capillary force. The cube sugar filled with the PDMS precursor is placed in a thermal oven and cured for 3 h at 90 °C. After the thermal curing process, the sugar grains were completely dissolved in deionized water and the water inside the PDMS sponge is fully dried (Fig. 1b). The dried PDMS sponge was alternately dipped with 10 mm min^{-1} into graphene ink (MExplorer Co., Ltd, Korea). The graphene-coated PDMS sponges are placed in thermal oven and cured for 30 min at 100 °C (Fig. 1c) to dry the ink and increase the adhesion force between the sponge surface and graphene ink. The graphene ink consists of dispersion of graphene in ethanol, dimethylformamide (DMF), and *N*-methyl-2-pyrrolidone (NMP), in particular, evaporation of ethanol in graphene dispersed ink results in a PDMS sponge with graphene attached on the backbone through strongly physical interaction, due to the CH–π interactions between the methyl groups of PDMS and the π—surface of graphene [22]. Figure 1d–i show the images of the surface of sugar, bare PDMS sponge, and graphene-coated PDMS sponge via field emission scanning electron microscope. Sugar grains stick together by evaporation of water (Fig. 1d, g). As shown in Fig. 1e, h, the PDMS sponge has a three-dimensional, interconnected, and micro-porous structure and the surface is clean. Figure 1f, i are SEM images of the surface of graphene-coated PDMS sponge, it is seen that the graphene is uniformly and thinly coated onto the surfaces of the micro-pores and formed a networks.
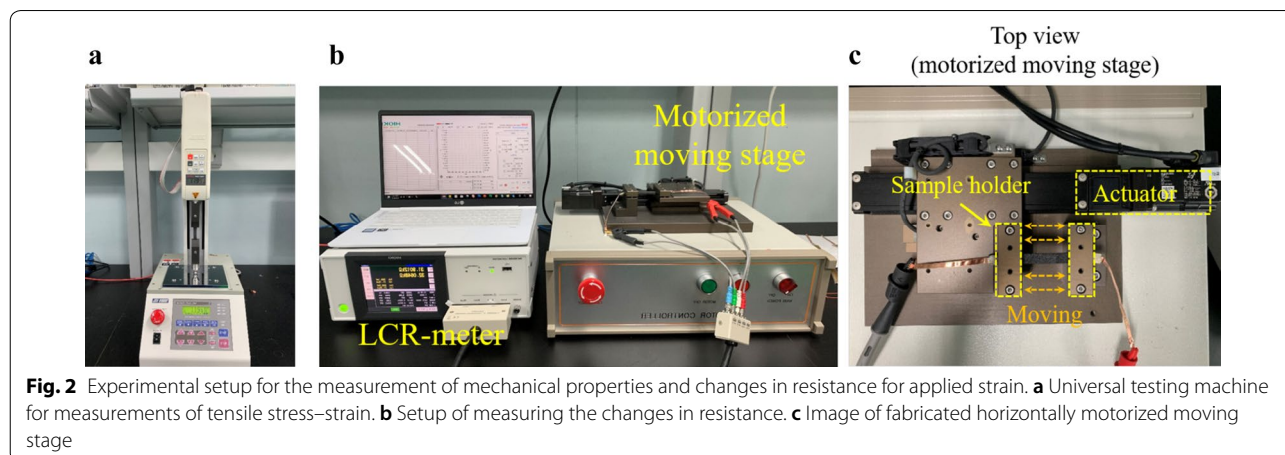
Measurement setup

The surface of the bare and graphene-coated PDMS sponges is characterized by FE-SEM (S-4800, HITACHI, Japan). Contact angle measurement was performed using

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Fig. 1 Photos of **a** cube sugar, **b** bare PDMS sponge, and **c** graphene-coated PDMS sponge. **d**–**i** Show the SEM images of surface of cube sugar, bare PDMS sponge, and graphene-coated PDMS sponge, respectively
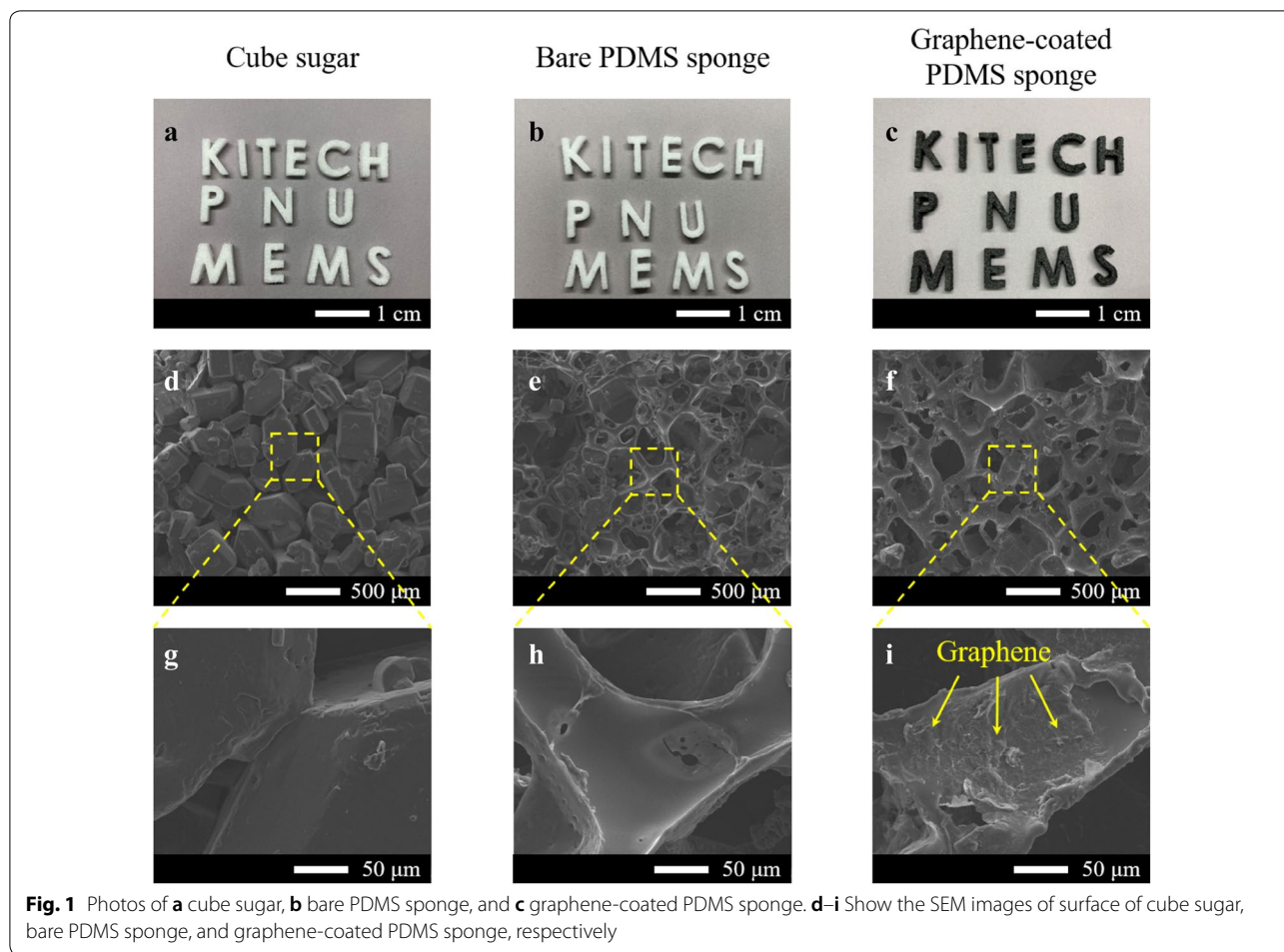
a contact angle analyzer (Phoenix 300 Touch, Surface Electro Optics, Korea) and a DI water droplet of 4 µL. For the measurement of tensile stress–strain curves of sponges, as shown in Fig. 2a, universal testing machine (JSV-H1000, JISC, Japan) was employed to evaluate the performance with load cell (HF-1, maximum load 10 N, load resolution 0.001 N). The sponges were stretched at a speed of 10% strain min^{-1} except strain rate test in mechanical evaluation. The test specimens for mechanical properties and strain sensors were prepared at

Fig. 2 Experimental setup for the measurement of mechanical properties and changes in resistance for applied strain. **a** Universal testing machine for measurements of tensile stress–strain. **b** Setup of measuring the changes in resistance. **c** Image of fabricated horizontally motorized moving stage

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40 mm (length, l) × 10 mm (width, w) × 3 mm (thickness, t) and 100 mm (l) × 10 mm (w) × 3 mm (t), respectively. An LCR-meter (HIOKI-3536, HIOKI, Japan) was used to measure the DC resistance of the sensors. All sensor evaluations were measured by connecting LCR-meter and computer in real time (Fig. 2b). We manufactured a tensile tester using a linear axis encoder (SRSH24YN-200, Misumi, Japan) and a linear actuator (LX2001CP, Misumi, Japan), controlled via Labview program (Fig. 2b, c). The voltage versus current characteristics were measured using a Parameter analyzer (Keithley 4200 SCS, Tektronix, USA). The copper tapes, which were the electrodes, were attached on two opposite sides of graphene wrapped sponge using silver conductive epoxy (CW-2400, ITW Chemtronics, USA).
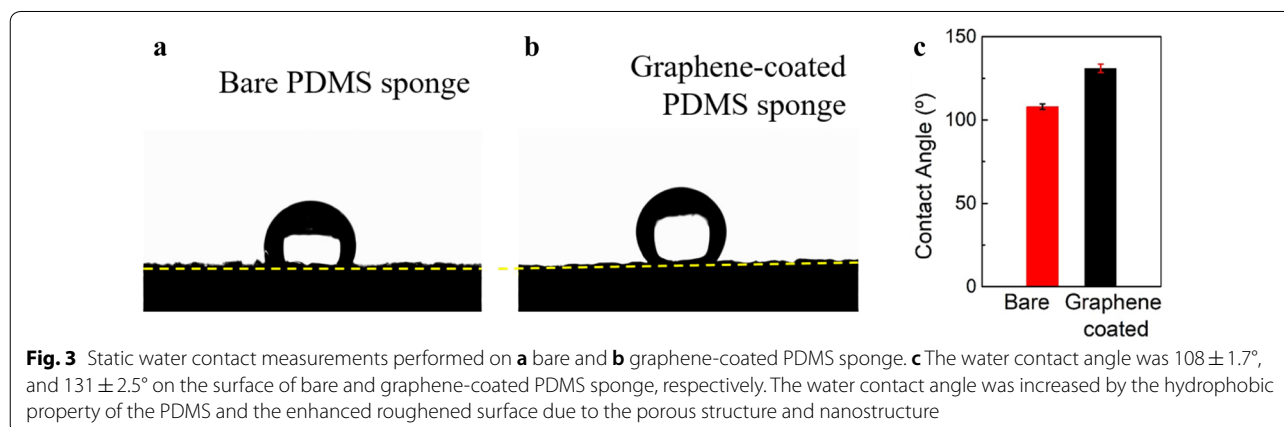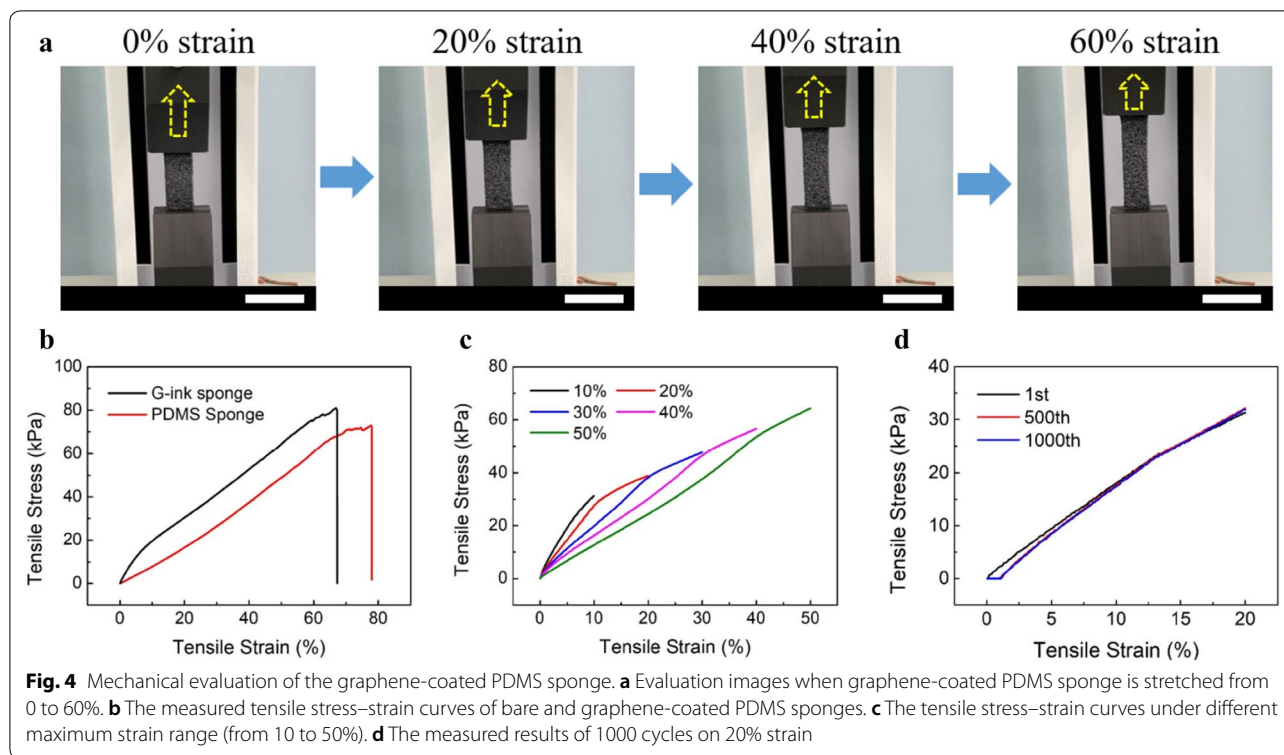
Results and discussion

The surface properties of bare and graphene-coated PDMS sponge was studied by contact angle measurement. Figure 3 shows the contact angle measurement results for the bare and graphene-coated PDMS sponges. The measured contact angle was the average obtained from five measurement results from each sponges at different points. The measured water contact angle was $108 \pm 1.7°$ on the surface of the bare PDMS sponge, and $131 \pm 2.5°$ on the surface of the graphene-coated PDMS sponge. PDMS is basically hydrophobic property with a water contact angle of 100°, which is further enhanced by a porous sponge that reduces the contact area between droplet and solid surface [30, 31]. Coating with graphene further increases the contact angle as 131° for the enhanced roughened surface due to nanostructured by of graphene [22]. The low standard deviation as 2.5 of measurement results also shows that the graphene ink is fully and uniformly coated on the surface of PDMS sponge.

The sponge is highly elastic and flexible, and it can be deformed with variation shapes without plastic deformation. To quantitatively evaluate the mechanical properties of bare and graphene-coated PDMS sponges, we measured the tensile stress–strain as a function of maximum strain range, strain range, and repeated cycles. Figure 4a shows an evaluation images when graphene-coated PDMS sponge is stretched from 0 to 60%. Figure 4b indicates the tensile stress–strain curves of two sponges until the failure. In cases of bare PDMS sponge, maximum tensile stress of about 72.9 kPa was required for 77.7% strain, while graphene-coated PDMS sponge required a pressure of 80.9 kPa for the 68% strain. The larger pressure required for the same deformation after graphene coating is due to the increased stiffness of the sponge with coating of graphene that is excellent mechanical properties. Although the tensile strain has been reduced due to the increased stiffness, it can be deformed up to 60% tensile range, making it suitable for a variety of wearable systems such as human motion detection [32]. Furthermore, we measured the tensile stress–strain under various strain (Fig. 4c). In can be seen that the maximum stress increases with increasing strain, and the curve is divided into two distinct stages. Figure 4d indicates the cycles test results. A 20% strain was applied to the strain sensors with 1000 cycles repeatedly, the permanent deformation occurred at the strain within 1%, but the tensile stress is almost similar.

The graphene-coated PDMS sponge can be used for wearable applications such as strain sensors because its conductivity, flexibility, and stability. When the sponge is stretched, graphene network changes along with the opening/closure of micro-pores, which causes the change of the conductive network. The sensing performance of the strain sensors based on graphene-coated PDMS sponge was measured under static loads. Figure 5a depicts the relative resistance variation of the strain sensor in a 40% strain range. The resistance variations were not significant up to 10% strain range, but it
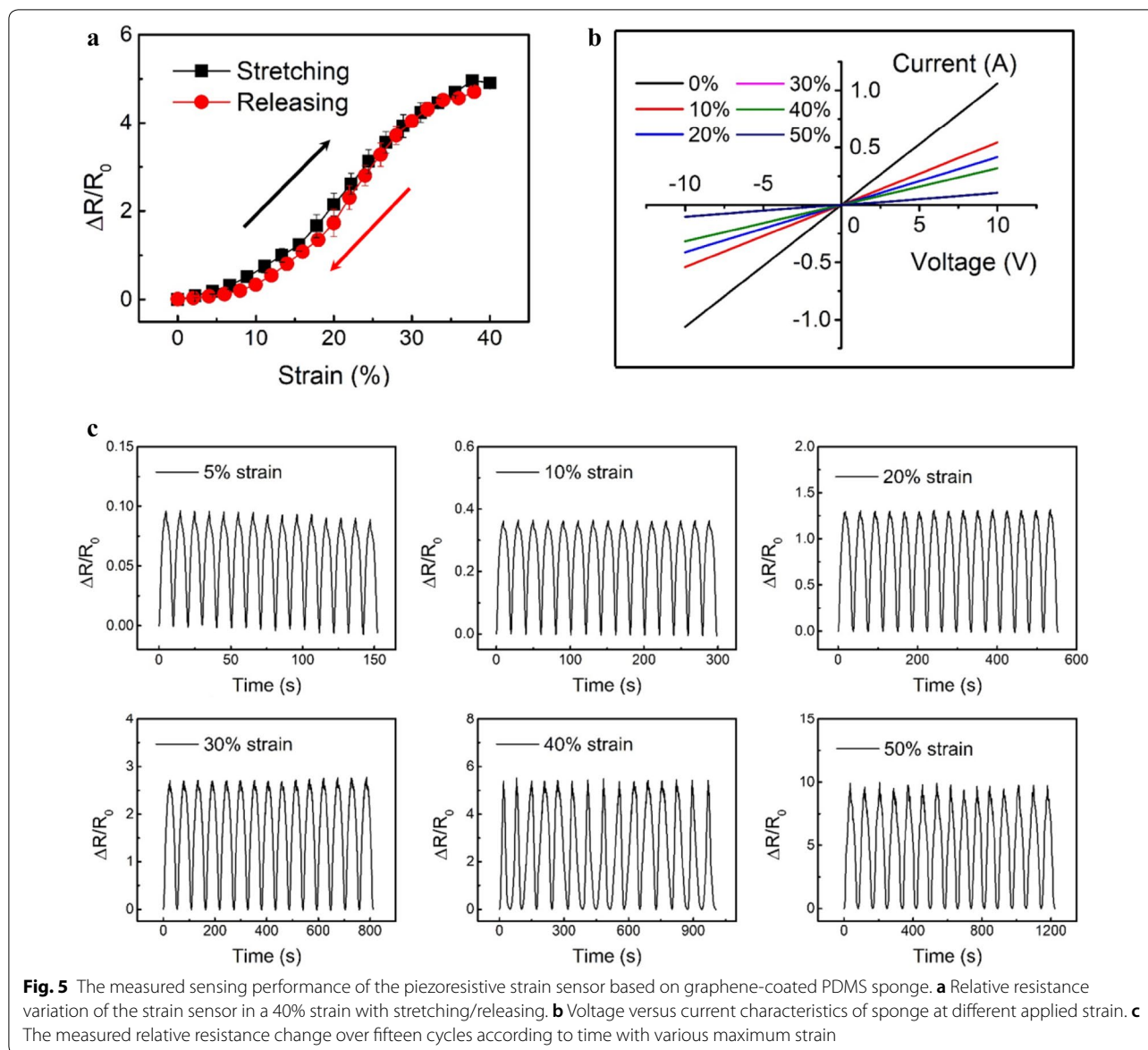

Fig. 3 Static water contact measurements performed on **a** bare and **b** graphene-coated PDMS sponge. **c** The water contact angle was $108 \pm 1.7°$, and $131 \pm 2.5°$ on the surface of bare and graphene-coated PDMS sponge, respectively. The water contact angle was increased by the hydrophobic property of the PDMS and the enhanced roughened surface due to the porous structure and nanostructure

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Fig. 4 Mechanical evaluation of the graphene-coated PDMS sponge. **a** Evaluation images when graphene-coated PDMS sponge is stretched from 0 to 60%. **b** The measured tensile stress–strain curves of bare and graphene-coated PDMS sponges. **c** The tensile stress–strain curves under different maximum strain range (from 10 to 50%). **d** The measured results of 1000 cycles on 20% strain

was confirmed that the increase rapidly in the 10–30% range. The stretching and releasing curves is almost overlap, sensors showed negligible hysteresis and this has been attributed to the reversible deformation of opening/closure of micro-pores. Figure 5b shows the voltage (V) versus current (I) characteristics of graphene-coated PDMS sponge at different applied strains. It is obviously observed the slope of the V versus I curves decreases with the applied strains. The slope on the V versus I curves is the resistance by Ohm's law, it means that the electrical resistance is dramatically changed by the applied strain. Figure 5c presents the relative resistance ($\Delta R/R_0 = (R - R_0)/R_0$, where R and R_0 denote the resistance values with and without applied strain) change over fifteen cycles according to time. The maximum strain is increased from 5 to 50% with same strain rate (10% strain min^{-1}), the minimum strain is fixed at 0%. In 5% strain, the $\Delta R/R_0$ decreases slightly from 0.096 in the first cycle and to 0.089 in the 15th cycle. However, after 10% strain, it can be seen that the $\Delta R/R_0$ are highly stable in all cycles. When the maximum strain is increased from 10 to 50%, the value of $\Delta R/R_0$ also increased by 0.363 to 9.604. Furthermore, there is stable signal in the relationship between the $\Delta R/R_0$ according to repeated cycles, indicating excellent reliability of the graphene-coated PDMS sponge in the various strain range. The sensitivity (gauge factor, $\delta(\Delta R/R_0)/\varepsilon$, where ε denotes the applied

strain) to strain at 10 to 50% was calculated to be 3.63, 6.43, 8.82, 13.09, and 19.21, respectively. It can be seen that the sensitivity of the strain sensor exponentially increases with increase of the maximum strain. The sensitivity exponentially increases by strain, theses high sensitivity of 19.21 and 50% strain range sufficient enable the strain sensors based on graphene-coated PDMS sponge to detect a variety of human body motion.
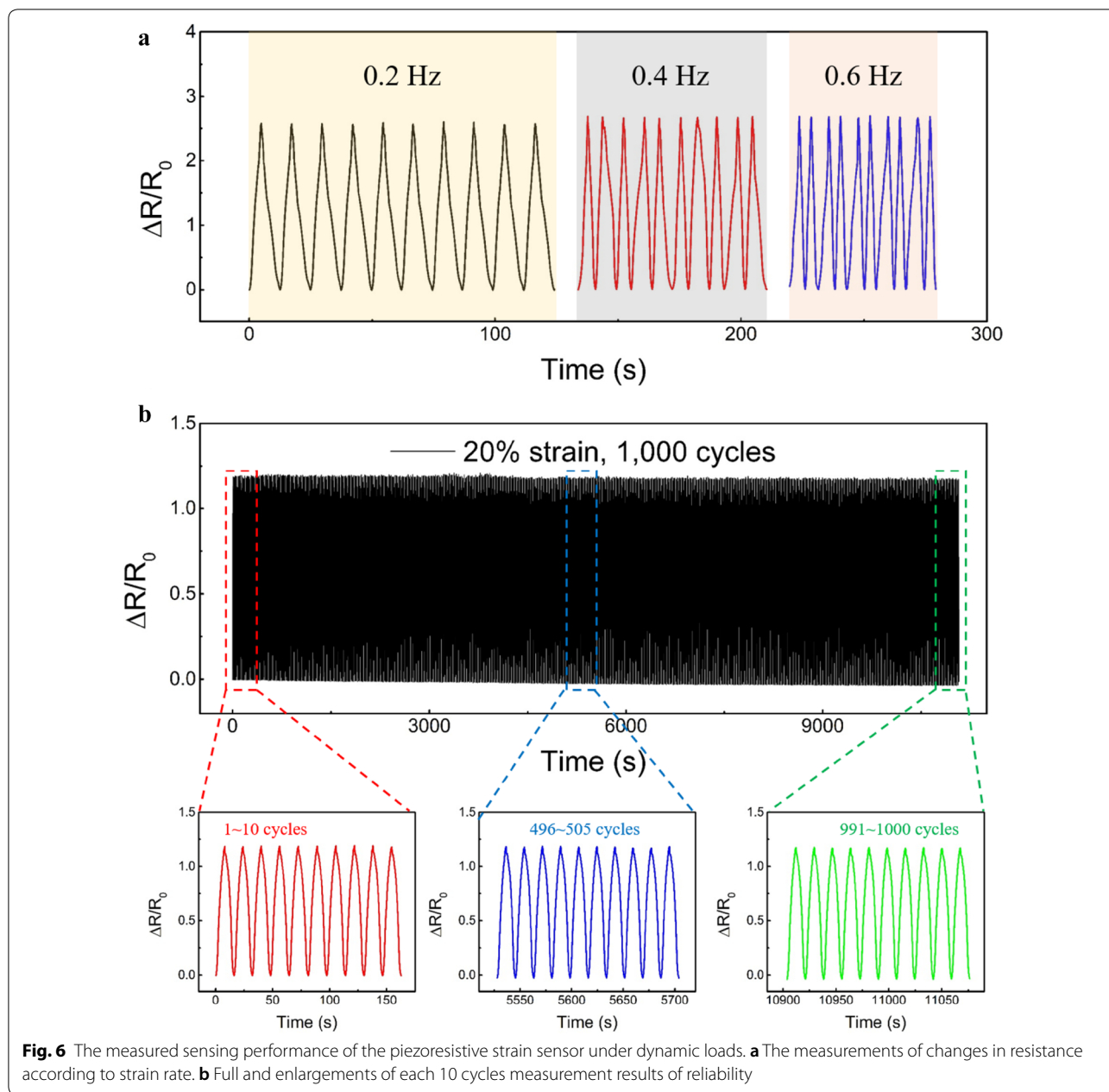
The flexible strain sensors are mainly used to detect deformation of the human body, knee and elbow deform rapidly and repeatedly. Therefore, it is important that the sensing performance have to maintain without any signal degradation in dynamic as well as static loads for real-time applications. Figure 6a shows the measurements of changes in resistance according to strain rate. The strain rate was varied from 0.2 to 0.6 Hz. The average resistance change was 2.558 at 0.2 Hz and 2.639 and 2.678 at 0.4 and 06 Hz, respectively. Although the resistance increased by 4.6% as the strain rate changed from 0.2 to 0.6 Hz, this means that the sensing performance is highly stable under diverse strain rates, it can be used to variety of applications. To investigate the durability of strain sensor, 1000 cycles of repeated stretching/releasing with a strain of 10% applied to the sensor at a 10% strain min^{-1} for 11,000 s. Figure 6b indicates the full and enlargements of each 10 cycles measurement results of reliability. The initial,

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Fig. 5 The measured sensing performance of the piezoresistive strain sensor based on graphene-coated PDMS sponge. **a** Relative resistance variation of the strain sensor in a 40% strain with stretching/releasing. **b** Voltage versus current characteristics of sponge at different applied strain. **c** The measured relative resistance change over fifteen cycles according to time with various maximum strain

middle, and last 10 cycles are shown in the red, green, and blue graph, respectively. The measured average resistance values were 1.186 in the initial cycles, 1.178 in the middle cycles, and 1.171 in the last cycles. The decreased change in resistance is only 1.3%, and these values can be negligible for practical applications. The reason why the graphene-coated PDMS sponge shows high stability and reliability is that the graphene bonds strongly to the backbone of PDMS due to the CH–π interactions, and the graphene does not peel-off even after repeated cycles. From these experimental results, we can verify that our strain sensors have good signal stability under static and dynamic loads against repeated stretching/releasing cycles.

Conclusion

In summary, we have developed a highly elastic and wearable piezoresistive strain sensor based on three-dimensional, micro-porous graphene-coated PDMS sponge, which is suitable for being attached on the curvature surface with several excellent properties in terms of the electrical and mechanical performance. The proposed strain sensors were easily produced by a sugar templating process and dip coating method based graphene dispersed ink without any additional process. The fabricated graphene-coated PDMS sponge was able to be deformed up to 60% strain and showed stable mechanical properties even under various strains and repeated cycles. Because of the changed conductive network between

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Fig. 6 The measured sensing performance of the piezoresistive strain sensor under dynamic loads. **a** The measurements of changes in resistance according to strain rate. **b** Full and enlargements of each 10 cycles measurement results of reliability

graphene-coated on the micro-pores, the strain sensor showed a high sensitive as 19.21 at 50% strain, with high repeatability and low hysteresis. Furthermore, we verified the strain sensor had stable sensing performance in various strain rates and high reliability in 1000 times repeated cycles. Finally, we expect our study will contribute toward the development of sensitive and wearable strain sensors that can be applied to various applications including electronic skin, soft robotics, health monitoring, and human–machine interfaces.

Authors' contributions
YJ and HCC developed the idea. KKJ, BGP carried out fabrication and literature search. YJ, JYC, and DHK contributed to measurement. YJ, JHP, and JSK drafted the manuscript. HCC supervised the research and reviewed the manuscript. All authors read and approved the final manuscript.

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