Abstract

Stretchable strain sensors have attracted much attention for health monitoring systems, human-machine interfaces, and robotics. However, there is still a challenge to develop strain sensors with excellent mechanical stretchability and high strain-sensing region as well as cost-effective fabrication process. Herein, a highly sensitive, reliable and low-cost strain sensors are developed using a resistive transduction of poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) and silver nanowire (Ag NW). Natural rubber was used as a stretchable substrate material, and a gold leaf was used as electrodes placed between PEDOT:PSS and Ag NW. The electrical resistance of this novel strain sensor shows an average value of 74.72 ± 14.65 Ω with a large sensing range up to 50% strain and sensing sensitivity of 418. The sensing response does not deteriorate after 750 stretching-releasing cycles and the sensor exhibits high stability after storage in air for more than 53 days. Concerning finger and joint movement, the wearable strain sensor demonstrates a stable output signal and distinguishes response under different bending and stretching. These advantages make it potential applications in wearable electronic devices and promising development in healthcare management.

Keywords

natural rubber, PEDOT:PSS, plasma treatment, silver nanowires, strain sensor
1 | INTRODUCTION

Wearable strain sensors are widely researched by a diverse set of materials in the past decade due to their advantages in many applications such as healthcare management,\(^1\) athlete motion detection,\(^2\) human-machine interface, and soft robotics.\(^3\) The resistive strain sensor has advantages in terms of its simple design, simple input/output control system, and low-noise ratios compared to other transduction mechanisms such as capacitive\(^4\) and piezoelectric.\(^5\) The sensitivity of resistive strain sensors is generally conducted by two main parts consisting of stretchable conductor and elastic substrate. To date, a variety of conductive materials have been reported for resistive strain sensors such as carbon nanotubes,\(^6\) graphene,\(^7\)–\(^10\) conductive polymers,\(^11,12\) metal nanowires,\(^1,13,14\) and metal nanoparticles.\(^15\) The classic conductive polymer, poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS),\(^11,16\) is attractive due to its high flexibility, good stretchability, and tunable electrical conductivity. However, PEDOT:PSS films on flexible substrates exhibited high resistance, small sensing regions and low sensitivity under tensile strains.\(^17\) Other types of strain sensors based on silver nanowire (Ag NW) have poor stability and weak adhesion on flexible polymer substrates, thereby causing a permanent loss of contact between adjacent Ag NW under numerous stretching-releasing cycles.\(^18\) Therefore, new strategies are required to fabricate strain sensors with high sensitivity, good stretchability, and stable performance.

The combination of advantages of each conductive material is a possible route for making high-performance strain sensors. For example, Ag NW/graphene-based strain sensor\(^19\) fabricated by casting polyurethane (PU) on the Ag NW/graphene hybrid particles and peeling-off from the membrane. The devices showed a high sensing response and strain resolution but a small sensing region (<1%). Chen et al. demonstrated an ultrahigh stretchable strain sensor using graphene/Ag nanoparticles synergic conductive networks.\(^20\) However, the stretchable strain sensor showed a low response in strain measurement range that was lower than 50%. Fan et al. reported PEDOT:PSS/Ag NW strain sensor by dipping PEDOT:PSS/Ag NW hybrid films into polydimethylsiloxane (PDMS) solutions followed by peeling-off.\(^18\) It endowed the device with an enhanced stretchability up to 50% due to the conductive PEDOT:PSS layer which compensated the Ag NW percolation networks for a large loss in conductance. However, the sensing response (ΔR/R\(_0\) ≈ 8 at 50% strain) should be improved further. In addition, a residual nitric acid in the fabrication process may affect long-term device stability and irritate the human body for wearable device applications. Therefore, there are still challenges to provide a stretchable strain sensor that possesses high sensitivity, good stretchability, and stable performance. Additionally, strain sensors with a simple design, nontoxic features and low-cost are desired for wearable device applications.

The sensitivity of sensors not only depends on the conductive materials but also depends on the stretchability of elastic substrates, for example, PDMS,\(^14,18,21,22\) PU\(^19,20,23\) and rubber.\(^6,9,10,24\) Natural rubber (NR) is an eco-friendly polymer elastomer with high strength, large elongation and good elasticity. NR surface characteristic allows a strain sensor to be easily attached to other materials leading to excellent adhesion to different parts of the human body. To the best of our knowledge, using NR as the base material for a strain sensor was an under-researched area.

In this work, we reported the performance of a strain sensor based on the resistance change of PEDOT:PSS and Ag NW hybrid films. NR was used as a stretchable substrate which was treated by microwave plasma to induce highly adhesive PEDOT:PSS films on a substrate. Using a gold leaf electrode as an inter-layer between PEDOT:PSS and Ag NW, the sandwiched films are robustly contacted and provide multi-pathway of electron transport along with the conductive films under stretching and releasing. This strain sensor showed stable performance with a high sensitivity of 418 and a broad sensing range up to 50% strain. In addition, its good response to bending and high working reliability allowed strain sensor to successfully detect human finger and joint movements, making it a promising candidate for applications in human motion detection and healthcare management.

2 | EXPERIMENTAL SECTION

2.1 | Materials

**Materials:** Silver nanowire (Ag NW) in isopropyl alcohol (IPA) was purchased from Nanopyxis, South Korea. The average length of the Ag NW is 30 μm. PEDOT:PSS aqueous solutions were purchased from Sigma-Aldrich.

**Natural Rubber:** The stretchable natural rubber (NR) substrate used in this work was received from S.K. Polymer Co., Ltd., Thailand. The NR sheets were made using a block rubber (Standard Thai Rubber, STR 5 L) mixed with some chemicals under a two-roll mill. Briefly, block rubber 100 parts per hundred of rubber (phr) was mixed with Wingstay L 1.5 phr, zinc oxide 5 phr, stearic acid 1 phr, and peroxide curing agent 2 phr. The final NR sheets with a size of 300 mm x 300 mm and 0.5 mm thickness were obtained.

2.2 | Methodology

**Preparation of sensors:** Figure 1 shows a schematic diagram of the fabrication process of a strain sensor along
with its bending and attaching on different surfaces. The natural rubber (NR) sheet, cut in a size of 35 mm x 35 mm, was attached on a glass substrate and cleaned by rinsing with deionized water, blowing with nitrogen gas, and followed by a microwave plasma treatment for 15 s four cycles. To prepare a pristine film, an aqueous solution of PEDOT:PSS 300 μl was spin-coated on the NR substrate with spin speed of 4000 rpm for 30 s, and then annealed on a hotplate at 60°C for 30 min. Next, a gold leaf electrode was prepared on the top of PEDOT:PSS film using mechanical rolling technique. Then 200 μl of 2% Ag NW solution was spun on the electrodes with spin speed of 1000 rpm for 20 s. After that, it was fully annealed at 60°C for 60 min. Finally, the hybrid film of Ag NW/PEDOT:PSS on NR substrate was peeled off from the underlying glass substrate and cut into a fixed size of 5 mm x 35 mm to study its electrical properties and strain sensing performance.

2.3 | Characterization

Characterization and measurement of strain sensors: The film morphology was characterized by optical microscopy (Olympus, Japan). Stress-strain curves were obtained using Dual-Range Force Sensor (Vernier, USA) integrated with motorized linear stage and screw side action tensile grip. In-situ conductivity-strain measurements were carried out using Keithley 2400 Source Meter with a two-probe method. The resistance change corresponding to the tensile strain was automatically measured and controlled using computer software. The relative resistance change (ΔR/R₀) was calculated based on the initial resistance R₀ and the change in resistance ΔR = R−R₀, where R is the resistance at a given strain value.

3 | RESULTS AND DISCUSSION

3.1 | Mechanical behavior of the natural rubber substrate

The mechanical response to the various deformations of the NR substrates was preliminarily studied both before and after plasma treatment of the NR surfaces. Figure 2 shows the stress–strain curves for NR substrates (size: 5 mm x 35 mm) measured along a monotonic strain ramp at a constant displacement rate of 15 mm/min. The six specimens show a good repeatability with the standard deviations of 5% as shown in Figure S1. The material stress at 100% strain was 0.54 MPa and 0.35 MPa for bare and plasma-treated NR surfaces, respectively. The results demonstrated the same order as the standard test methods for rubber (ASTM-D412-16), whose values are reported in Table S1.

The mechanical properties of the NR substrate with strain ramp tests were evaluated and displayed in Table 1. As shown in Figure 2(A), the NR demonstrates a linearly increasing trend at low level strains of stress–strain curves, which its slope refers to the modulus of elasticity or Young's modulus. At higher levels of strain, the reductions of the slope value were observed, and the
The yield phenomenon was evaluated from the deviations between the first and the second linear trends. The yield stress of the plasma-treated NR (0.18 MPa) is slightly lower than that of un-treated NR (0.20 MPa). However, the yield strain has been enhanced by the plasma treatment in which NR undergoes yielding and localized deformation at strain levels of ~28%. Therefore, to assure a reliable stretching-releasing signal in the elastic region, the maximum applied strain could not exceed 30% with a minimum value of 0.02%.

A cyclic stretching-releasing test was carried out to study the substrate response under the strain history. Figure 2(B) and Figure S2 show the output of the cyclic test up to 100 cycles of plasma-treated NR. The hysteresis response (Figure 2(B)), which was confirmed by the area in the stress–strain correlation, exhibits a progressive reduction as the number of repeated cycles increases. The results indicate that the rubber undergoes a large evolution of stress–strain characteristics in the first cycle, and tends to a common trend after 5 cyclic stretching-releasing tests. Furthermore, the mechanical deformation at the end of each cycle slightly decreases as increase the repeated stretching-releasing cycle. At 60% strain, the results reveal that the mechanical deformation reduces 0.8% after the first cycle and approaches 8.3% after 100 cycles. These results show that the natural rubber is suitable as a stretchable substrate for the strain sensor.

### 3.2 Properties of the strain sensor

The strain sensors were fabricated using a layer-by-layer coating of stretchable conductor on NR substrate. A layer of PEDOT:PSS was spin-coated on the substrate followed by attaching gold leaf electrodes and coating Ag NW on the top layer, as displayed in the fabrication process in Figure 1(A). The fabricated strain sensors were bendable and directly attached on human skin or onto a curved surface (Figure 1(B–D)). The plasma treatment can improve the adhesion of PEDOT:PSS on the NR surface as shown in the results of our previous report. Uniform PEDOT:PSS films have a base resistance of 4.02 ± 0.51 MΩ (size: 5 mm x 35 mm). However, the sensor resistance dramatically decreased to 74.72 ± 14.65 Ω after being coated Ag NW, due to its high conductive and percolation nanowire networks. The resistance distribution curve of Ag NW/PEDOT:PSS/NR strain sensors were shown in Figure S3.

Figure 3(A) illustrates the current–voltage (I–V) characteristics of the Ag NW/PEDOT:PSS/NR strain sensor with un-loading and loading tensile strains of 5% and 10%. It is obvious that all the I–V curves are linear characteristics, indicating ohmic behavior of the sensor. The conductivity of the devices is determined by a competitive channel of PEDOT:PSS, Ag NW and their complex network. The slope of I–V curve gradually decreased with the increase of strain, suggesting that the resistance of sensor increased under strain elongation.

The strain-dependent resistance of Ag NW/PEDOT: PSS/NR sensor as a function of continuously applied strain is shown in Figure 3(B). It can be seen that the resistance of the sensor slightly increases at a low applied strain, and abruptly increases in magnitude until failure. The maximum strain happens at ~50% which two times greater compared to the Ag NW/NR sensor. These sensing capabilities of the sensors are limited by the substrate
and conductive layer ductility. The Ag NW sensors showed a small sensing region probably due to a high cracking or slipping of Ag NW networks which cause discontinuous conduction pathways under the larger strains. At a lower tensile strain of 5%, the Ag NW sensor showed a highly reliable resistance response, but it failed to be recoverable after full relaxation especially for repeatedly stretching-releasing tests with higher applied strains (Figure 3(C)). Moreover, the resistance responses show a shift up of initial value for each testing cycle. The resistance increase may be caused by the cracking of Ag NW generated discontinuities of nanowire networks, leading to irreversible deformation in the testing process. However, by integrating with the conductive PEDOT:PSS layer, a large loss of conduction pathway due to a cracked or slipped of Ag NW networks was compensated. As shown in Figure 3(D), the Ag NW/PEDOT:PSS strain sensor demonstrated the extending detection ranges with a good reversible response under dynamic stretching and releasing cycles. The final resistance was slightly increased by ~10% after ten cycles of stretching-releasing strains, demonstrating highly reliable and repeatable devices, and probably a great potential for practical applications.

To investigate the sensing mechanism of the strain sensor in microscopic view, the surface morphology of the Ag NW/PEDOT:PSS coated on the NR substrate was monitored under different tensile strains. As shown in Figure 4(A), there is no crack or wrinkle formation in the conductive layer coated on the NR substrate for the as-prepared device. Under an applied voltage, electrons can pass through overlapped Ag NW (low resistance channel) within the percolation networks easier than PEDOT:PSS film (high resistance channel), corresponding to a low level of initial resistance after coated Ag NW on PEDOT:PSS layer (Figure 4(B), left). When the strain sensors were stretched to 10% strain, small-domain microcracks in the perpendicular direction of stretching appeared on the film. At this sensing region, the increase in electrical resistance might be the cracking and repositioning of nanowires led to smaller overlapped areas and larger interspacing between nanowire networks. However, the main conductive channel still depended on the high conductive channel of Ag NWs showing small resistance change as indicated in Figure 3(B). The density of microcracks increased when the sample was further stretched (20% strain), which resulted in more disconnection among Ag NW networks and led to a further increase in

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the electrical resistance. A large loss in conductance of Ag NW networks was compensated by the PEDOT:PSS layer, electrons can pass through the Ag NW and PEDOT:PSS interfaces instead of discontinuities of Ag NW networks (Figure 4(B), middle). When the strain was increased to 30% and 40%, the surface morphology of the sample showed a large area of wrinkle domains, leading to a sharp increase in resistance of the sample under stretching states. This phenomenon can be attributed to the physical disconnection of nanowire networks and microcracks on PEDOT:PSS thin films leading to the conductive channel mostly conducted by a high resistance of the PEDOT:PSS layer and their interfaces with Ag NW (Figure 4(B), right). The significant contribution of microcrack generation on the resistivity of strain sensors was confirmed by measuring electrical signals of the PEDOT:PSS/NR sensors under strains (Figure S4). The results exhibit slight changes in resistance for the lower sensing region of 30% strain, and abrupt increase in resistance when the sample was stretched in larger strains.
Figure 5(A) shows a relative resistance change (ΔR/R₀) of Ag NW/PEDOT:PSS/NR sensors as a function of applied strains (ε). The sensing sensitivity or gauge factor, GF = (ΔR/R₀)/ε, can be evaluated from the slope of the ΔR/R₀ and strain curve. As shown in Figure 5(A), there are two linear trends which is the lower sensing region has a gauge factor of 25.75 (R² = 0.9917) and becoming abrupt increase up to almost 418 (R² = 0.9879) afterward. The limit of detection was ~3.5% strains. At the strain level of 30%, the first linear trend gradually approaches the second trend. Interestingly, the change in the sensitivity (slope) occurred near the yielding point of NR substrate (stress vs. strain curve). At lower strains, a conductive track was dominated by a low resistance of Ag NW networks and their interface junction with PEDOT:PSS films, therefore a small change in electrical resistance was observed in this region. In addition, the NR substrate is in the elastic region (Young's modulus), in which Hooke's law is valid within this limit, leading to high reliability in resistance response under stretching-releasing strains. However, as the strain increased to above 30% strain, not only was there high microcracking of Ag NW and detachments of their interfaces but there was also microcracking of the PEDOT:PSS layer leading to a large increase in resistance under the small change in applied strain. The comparison between our results and other works can be found in Table S2 which shows strain range and gauge factor on materials related to PEDOT: PSS/Ag NW and Natural Rubber.

For stretchable sensors, the bending sensitivity behavior is one of its important properties. Here, a continuous bending angle was applied to the sensor by designing a concise way to define them as applied strain (Figure 5(B)). It was found that the relative resistance increases linearly with the degree of bending in the range of 0–90° and an almost repeatable response in the released direction. The results indicated a crucial application for the sensors, especially in biomedical-based body movement. For the practical application of strain sensor devices, the long-term stability of the sensor was evaluated. The Ag NW/PEDOT:PSS/NR samples were placed in the air at room temperature and their resistances were recorded over a period of 53 days as shown in Figure 5(C). The results show a highly stable initial resistance of the devices which resistance increase ~10% after 30 days. The repeatability of the Ag NW/PEDOT:PSS/NR strain sensor has been investigated under the 750 cyclic stretching-releasing test at 10% strain, as shown in Figure 5(D). We found that the sensor response can be mostly returned to the initial conditions under
stretching-releasing test. In addition, the sensor response slightly increases in the range of 15% after 750 cycles stretched, demonstrating good repeatability and reliability response due to the robust structural integration of good viscoelasticity of natural rubber, highly conductive Ag NW and PEDOT:PSS.

3.3 Monitoring the human joint motion

Due to the high sensitivity, good stretchability, and excellent stability of the Ag NW/PEDOT:PSS NR strain sensors, their applications were conducted by detecting human motions. Figure S5 shows an experimental set-up for applying the strain sensor to detect the movement of human joints. The sensor as a resistive device was connected to the load resistor, and the output signal was recorded on the computer via a wireless signal by a microcontroller processing unit. By using this simple voltage divider circuit, the output voltage will decrease as strain-dependent resistance increases, and we can extract the resistance change of the sensor. The stain sensor was mounted on the index finger to detect the bending motions (inset in Figure 6(A)). The device can distinguish the deformation under diverse bending angles from 0 to 90° with good response reliability. In Figure 6(A), the sensing variation was detected obviously when the finger was bent at ~0, ~20, ~40, ~60, and ~90° and back to the initial state. With the step-by-step increase of the bending angles, the relative resistance change visibly rose, demonstrating reliable monitoring of the diverse deformation.

To further evaluate the reliability of the strain sensors, the continuous repeatability bending angles of ~45 and ~90° and a rapid bending at ~60° were observed, as shown in Figure 6(B). The sensors exhibit an accurate response to rapid bending of the finger and show outstanding stability. In Figure 6(C), the stretchable strain sensor was attached to a wrist and slow bending-straightening of wrist movement was monitored. Our sensor can discriminate the movement patterns of the wrist. Similarly, by attaching the sensor to the nape of the neck (Figure 6(D)), we can also detect head movements. These results prove that the Ag NW/PEDOT:PSS/NR strain sensors are promising candidates for further applications in human motions and medical healthcare management.

4 CONCLUSION

A high performance resistive strain sensor was prepared using natural rubber as a stretchable substrate, PEDOT:PSS and Ag NW as a flexible conductor. The fabrication was based on spin-coating the PEDOT:PSS on the plasma-treated NR surface followed by attaching gold leaf electrodes and coating Ag NW on the top layer. The Ag NW/PEDOT:PSS/NR strain sensor exhibits electrical resistance of 74.72 ± 14.65 Ω with excellent stability for 53 days of storage in air. The resistance change of the strain sensor depends on the multi-pathway of electron transport both of Ag NW networks and PEDOT:PSS layer as well as viscoelasticity of NR substrate. Thus, the fabrication sensors have a high sensing sensitivity up to 418 and large sensing region of 50% strain. Moreover, the devices exhibited high reliability after 750 cycles of stretching-releasing of 10% strain and good reversibility under the bending test. These strain sensors are successfully used in the detection of human finger, wrist, and head movements. We hope that this work will open a channel to use natural rubber as a stretchable substrate for electronic devices in the future.

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CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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