Highly Stretchable and Mechanically Stable Transparent Electrode Based on Composite of Silver Nanowires and Polyurethane−Urea

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S *Supporting Information*

ABSTRACT: Transparent electrodes based on conventional indium−tin oxide (ITO) can hardly meet the requirements of future generations of stretchable electronic devices, including artificial skins, stretchable displays, sensors, and actuators, because they cannot retain high conductivity under substantial stretching and bending deformation. Here we suggest a new approach for fabricating highly stretchable and transparent electrodes with good stability in environments where they would be stretched repeatedly. We designed polyurethane−urea (PUU), a urethane-based polymer, to enhance the adhesion between Ag nanowires (AgNWs) and poly(dimethylsiloxane) (PDMS). The adhesion could be further improved when irradiated by intense pulsed light (IPL). After delicate optimization of the layered AgNW/PUU/PDMS structure, we fabricated a stretchable transparent electrode that could withstand 100 cycles of 50% stretching−releasing, with exceptionally high stability and reversibility. This newly developed electrode is therefore expected to be directly applicable to a wide range of high-performance, low-cost, stretchable electronic devices.

KEYWORDS: stretchable electrode, Ag nanowire, intense pulsed light, polyurethane−urea, transparent electrode
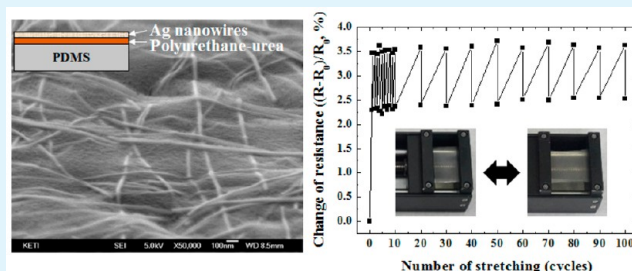
INTRODUCTION

Stretchable transparent electrodes have recently been gaining more and more attention because of their potential application in various implements including electronic skins and wearable and shape-deformable electronics.[1−5] The well-known brittle nature of conventional oxide-based electrodes, e.g., indium−tin oxide (ITO) electrodes, has spurred the research community to develop new fabrication approaches and transparent conductive materials that could be stretched without losing conductivity.[6−8] Therefore, there have been enthusiastic searches for more flexible and stretchable alternatives to ITO that can provide excellent transparency, low sheet resistance, and sufficient mechanical stability in stretching applications. Of the various alternatives to ITO, silver nanowires (AgNWs) have emerged as the most promising candidate by virtue of their good conductivity and percolated network structure.[9−11] They also offer the advantage of being easily formed into a highly stretchable and transparent conductive film through a simple and readily scalable solution-processing technique, which consists of spin coating, dip coating, and air spraying of the AgNW dispersion.

To attain stretchability of the transparent electrode through the use of AgNWs, a free-standing polymer also needs to be developed as a stretchable transparent substrate. Poly-(dimethylsiloxane) (PDMS) is considered as one of the powerful candidates because of its high transparency, neutral coloration, high elasticity, large elongation up to 160−180%, and biocompatibility.[12,13] There are already several reports on using the combination of PDMS and Ag fillers to form stretchable electrodes by various means.[14−18] Among these approaches, we focused our attention on two interesting methods: in the first method, a bulky mixture is prepared by adding Ag particles to the non-cross-linked PDMS liquid;[14] in the second method, percolated AgNW networks are buried at the surface of the cross-linked PDMS films.[15−18] The first method yields a three-dimensionally conductive electrode that could be used to fabricate stretchable printed circuit boards and other flexible electronic devices. However, the second approach is more beneficial because the unique structure of AgNW networks allows the use of only a small fraction of percolated AgNW networks to form a highly conductive electrode.

Regardless of the method employed, it is most important to ensure that the AgNWs are well adhered to the PDMS in order to construct an electrode with enough stability to endure repeated stretching and releasing. However, the hydrophobicity of PDMS hinders wetting of the PDMS surface by AgNWs, resulting in poor mechanical stability of the fabricated electrodes.[19] According to Akter and Kim, a coating of polydopamine on PDMS could change the surface from hydrophobic to hydrophilic so that AgNWs would be well adhered to the surface of cross-linked PDMS.[20] However, the time required to form a coating is too long (24 h), and the

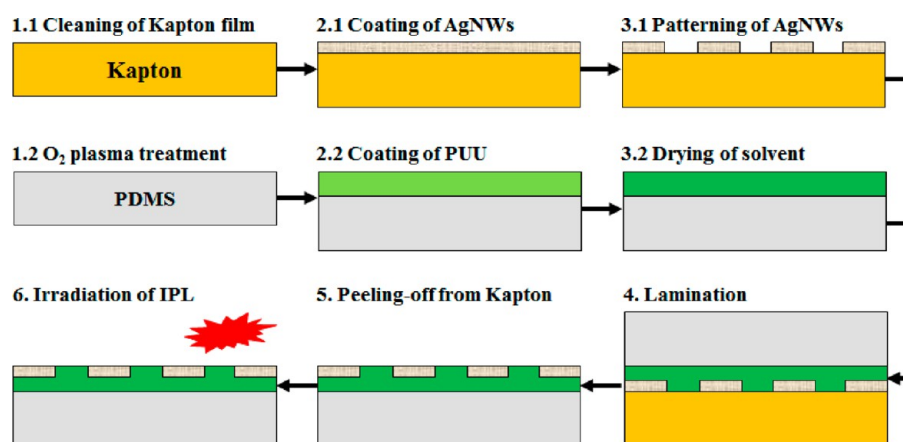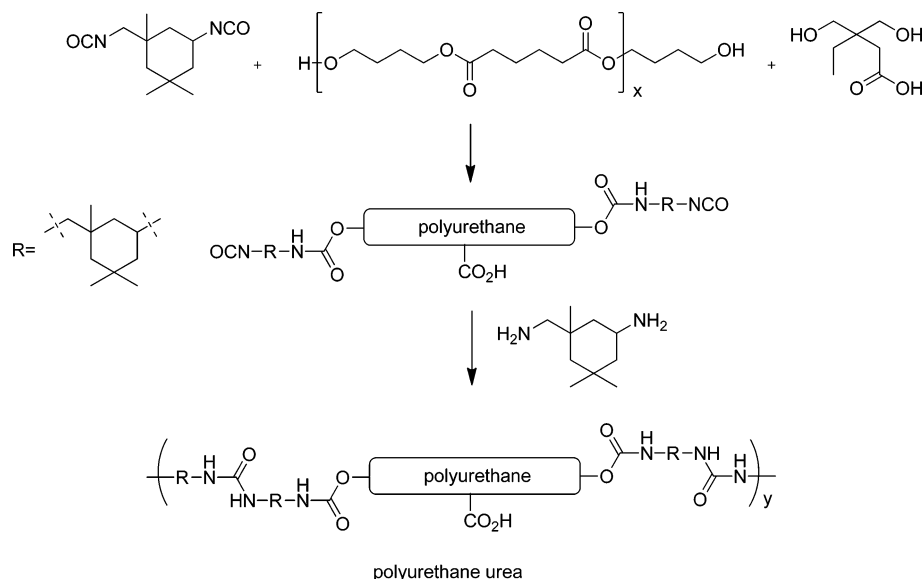**Scheme 1. Synthesis of Polyurethane−Urea**

Figure 1. Schematic of the fabrication procedure for patterned stretchable electrodes based on the AgNW/PUU/PDMS structure.

capability of the fabricated transparent electrode to withstand the elongation is not sufficient. They reported that the transparent electrode (80% transmittance) failed to maintain its conductivity when it was stretched by about 20%. Other studies employing AgNWs and only PDMS reported that a high AgNW content is needed to achieve the stretchable conductivity possibly owing to the loose adhesion between the two materials.[15−18] The electrodes prepared through those approaches, therefore, were no longer transparent, and their mechanical stability was scarcely discussed. This indicates that it is necessary to develop more stable materials that are highly compatible with AgNWs and to devise a simple method to combine them with PDMS.

In the study reported here, we synthesized polyurethane−urea (PUU) as a buffer material to enhance the mechanical stability of the AgNW−PDMS stretchable electrode. PUU displayed stronger hydrogen bonding than polyurethane (PU) because of the two hydrogen donors of the urea moieties.[21] Compared to the modulus of PU, the modulus of PUU exhibited a broader rubber plateau, which is a feature of an elastomer, because of the hydrogen bonding. In addition, the adhesion of PUU on AgNWs was improved by employing the carboxylic acid groups in its backbone,[22] and the favorable

interactions between AgNWs and the hydrogen donors increased the affinity of AgNWs to the polymer matrix. Using an inverted processing approach, percolated AgNW patterns formed on a separate substrate were successfully transferred to the surface of PUU that was preformed on a PDMS layer. We also employed simple irradiation with intense-pulsed-light (IPL) to increase the stability of the AgNW/PUU/PDMS electrode. Owing to the thermoplastic property of PUU, adhesion of the AgNWs to PUU could be further enhanced by partial melting of the underlying areas, resulting in embedment of the nanowires. Cyclic stretching up to a strain of 50% was carried out to evaluate the performance of the electrode.

■ EXPERIMENTAL SECTION

Materials. Polyester diol (number-average molecular mass, $M_w \sim$ 1000) was purchased from Songwon, Korea. 2,2-Bis(hydroxymethyl)-butyric acid and 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 is summarized in Scheme 1. The mixture of polyester diol (82.8 g, 82.8 mmol), 2,2-bis(hydroxymethyl)-
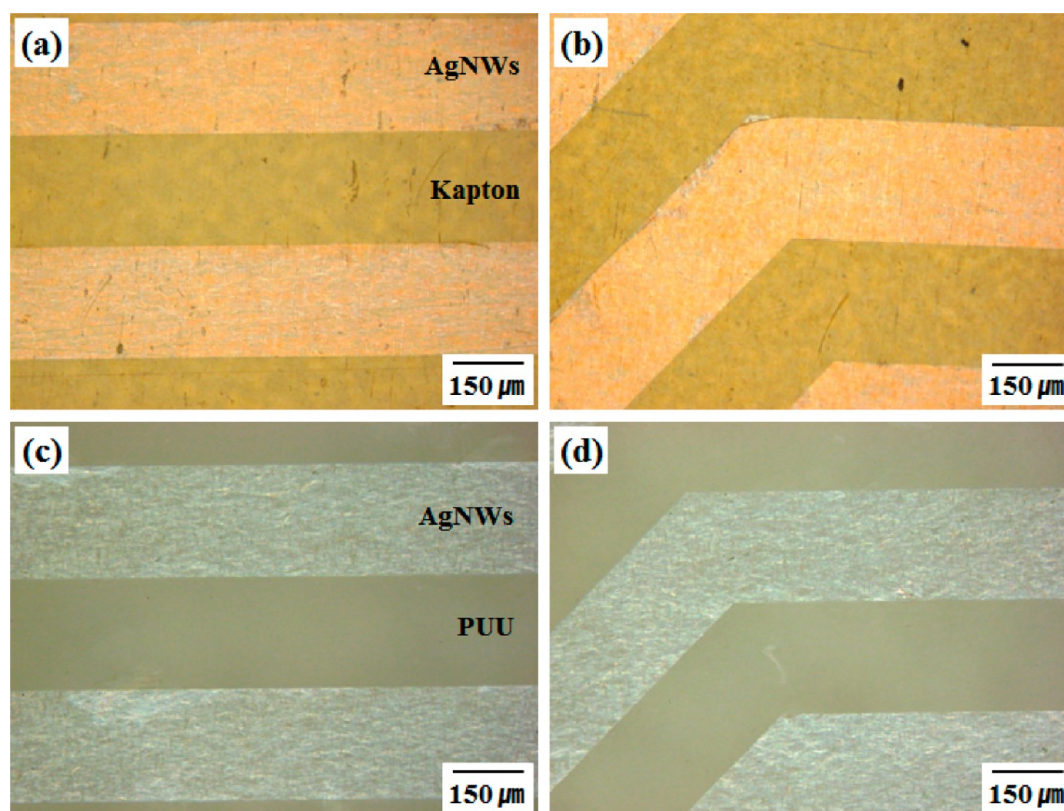
Figure 2. (a, b) AgNWs patterned on a Kapton film. (c, d) AgNWs transferred to the surface of PUU coating on PDMS.

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. After the solution was cooled to room temperature (RT), toluene (60 g) was added, and the viscous solution was heated at 70 °C. To this solution was added the 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. The addition of the mixture of toluene (28.8 g) and 2-propanol (14.4 g) afforded the solution a solid content of 25%.

Fabrication of Electrodes. The procedure used for the fabrication of the AgNW/PUU/PDMS transparent electrode is schematically illustrated in Figure 1. A polyimide (Kapton, DuPont, USA) film was first cleaned using detergent, deionized water, and isopropanol. The film was placed on a Mayer rod coater and several drops (0.5 mL) of a Ag nanowire solution (Nanopyxis Ltd., Korea) were deposited on it. The average diameter and length of the AgNWs were 40 nm and 30 μm, respectively. Immediately afterward, a Mayer rod #8 (R.D. Specialties, Inc., USA) was rolled over the drops to spread evenly the nanowire solution over the film surface. The resulting film was heated in an oven at 50 °C for 30 min to remove any remaining organic solvent from the coating layer. Photolithography was carried out to form the barriers for AgNW patterning, after which the AgNWs were etched away by an acidic solution (chromium etchant, Sigma-Aldrich, USA), followed by stripping of the protoresist (AZ1512, Merck, Germany). The point-to-point measurement of resistance revealed that the electrical performance was not deteriorated (not exceeding 10%) by the patterning process. In a separate procedure, a PDMS film was fabricated using the elastomer kit (w/w = 1:10).

Oxygen plasma treatment was employed to form hydroxyl functional groups on the PDMS film. The synthesized PUU was then coated on the PDMS with varying thickness by spin coating, followed by solvent drying. The patterned AgNW electrodes on the Kapton film were adhered upside down to the PUU/PDMS layers by a roll laminator that was heated to 80 °C. After cooling, the AgNW/PUU/PDMS film was peeled off from the Kapton film. A photonic sintering system (Sinteron 2000, Polytec Ltd., USA) was employed for

the IPL irradiation (input voltage: 1.8 kV) of the samples, with the exposure repeated for a pulse duration of 500 μs every time. The irradiation condition was optimized to enhance the adhesion and embedment of AgNWs to the PUU/PDMS film for maintaining conductivity while repeatedly stretched and released.

Evaluation. Field-emission scanning electron microscopy (FESEM; JSM6700F, JEOL Ltd., Japan) was used to investigate the microstructure of the AgNW networks. Their optical transmission was also measured using a UV−visible spectrophotometer (V-560, Jasco, Japan), whereas the sheet resistance (R_s) was measured by a noncontact measurement system (EC-80P, Napson Corporation, Japan). The surface morphology was measured by atomic force microscopy (AFM; XE-100TM, Park Systems, USA). An automatic stretch-testing machine (Stretching tester, Jaeil Optical System, Korea) was used to measure the long-term reliability under repeated cycles of stretching. The electrodes were elongated at a displacement rate of 5 mm/s, and their resistance was measured during both stretching and releasing. More than 10 samples were fabricated and measured for most parameters.

■ RESULTS AND DISCUSSION

PUU was synthesized employing a polyester as a soft segment and an isophorone moiety as a hard segment (the thermal behavior of the PUU is provided in Figure S1 of the Supporting Information). The physical cross-linking induced by the hydrogen bonding between N—H and C═O was enforced because of the urea moieties.[23] The incorporation of 2,2-bis(hydroxymethyl)butyric acid into PUU was expected to enhance the affinity of AgNWs for PUU.[22] As the AgNWs were surrounded by poly(vinylpyrrolidone) (PVP), the carbonyl groups of PVP presumably formed hydrogen bonds with the carboxylic acid groups of PUU.

As described in Figure 1, the PDMS film was treated with an O_2 plasma to produce the dangling hydroxyl groups on the
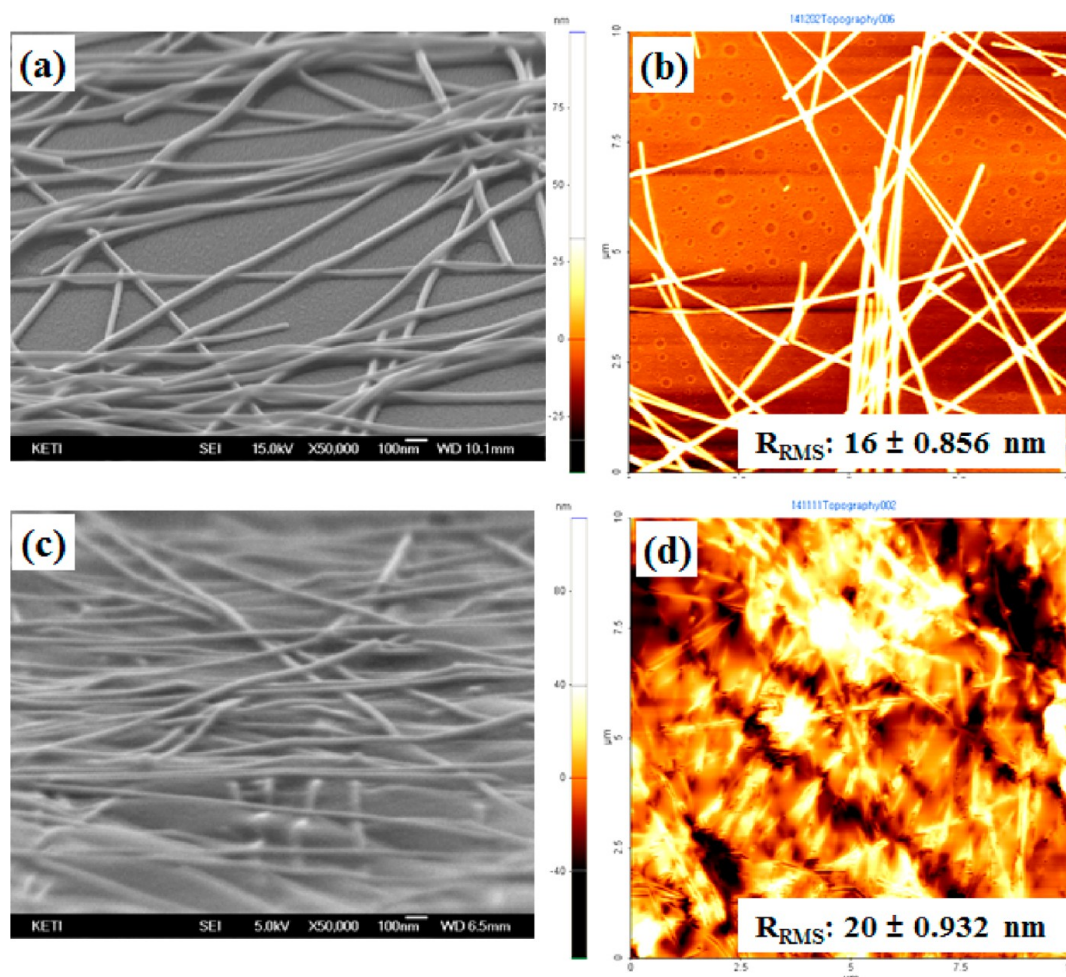
Figure 3. Topography of AgNWs on Kapton and PUU: (a, b) AgNWs on Kapton, (c, d) AgNWs on PUU. Panels a and c are FESEM images, whereas panels b and d show the morphologies obtained by AFM.

surface.[24] The surface recovered otherwise to become hydrophobic[25] was coated with PUU, where hydrogen bonding between the hydroxyl groups and urea (or urethane) was expected to enhance the interlayer interaction. After drying at 50 °C for 30 min, the PUU was impregnably adhered to the PDMS so that it would be impossible to segregate it from the underlying layer by any physical means. The AgNW networks that were prepatterned on a Kapton film were transferred to the PUU surface. For evaluation of the transferring efficiency, we compared the shapes of the patterns on Kapton (mother substrate) and PUU/PDMS (target substrate), as shown in Figure 2. By incorporating 2,2-bis(hydroxymethyl)butyric acid into PUU, the AgNW patterns were perfectly transferred to the PUU/PDMS structure. The pattern edges were sharp, and neither defects nor a gradient in AgNW density could be found. It is also notable that R_s of the AgNW networks increased by less than 10% after the transfer. Considering that the rolling-based lamination process is already popular in the electronics industry, the fabrication scheme developed here can be easily and practically adopted.

Figure 3 shows a comparison of the morphology of the AgNW networks on Kapton and PUU based on images obtained by FESEM and AFM. AFM measurements on bare PDMS and PUU are also shown in Figure S2 of the Supporting Information for comparison. As shown in Figure 3a, the nanowires were deposited on the flat surface of Kapton, while

the uncovered areas were bare, which resulted in a rough surface morphology. This would present a problem if the AgNW/PUU/PDMS ensemble is used as a substitute for an ITO bottom electrode of organic devices, e.g., organic light-emitting diodes and solar cells, because the rough surface could easily penetrate the soft organic layer and short circuit the device. This high roughness was also verified by the AFM measurement shown in Figure 3b. After the AgNWs were transferred to the surface of PUU, their structure was severely altered, as shown in Figure 3c. The nanowires were well attached to the PUU without any discernible gaps between them. However, despite the dense structure of AgNW/PUU/PDMS, the roughness remained high, as shown in Figure 3d. Considering that the roughness was not largely varied by the transfer process when AgNWs were not employed (see Figure S3), this can be attributed to the following factors: (i) even though compressional force was applied during the lamination, the nanowires were merely adhered to the PUU and they did not penetrate the polymer matrix; (ii) the force should be sufficient to exceed the initial adhesion between AgNWs and the Kapton film to properly transfer the AgNWs. The forces used to peel off the AgNWs from the Kapton induced local strains in the PUU and eventually formed the ripples on its surface.

Encouraged by the fact that the synthesized PUU is a thermoplastic material, we employed IPL to generate further
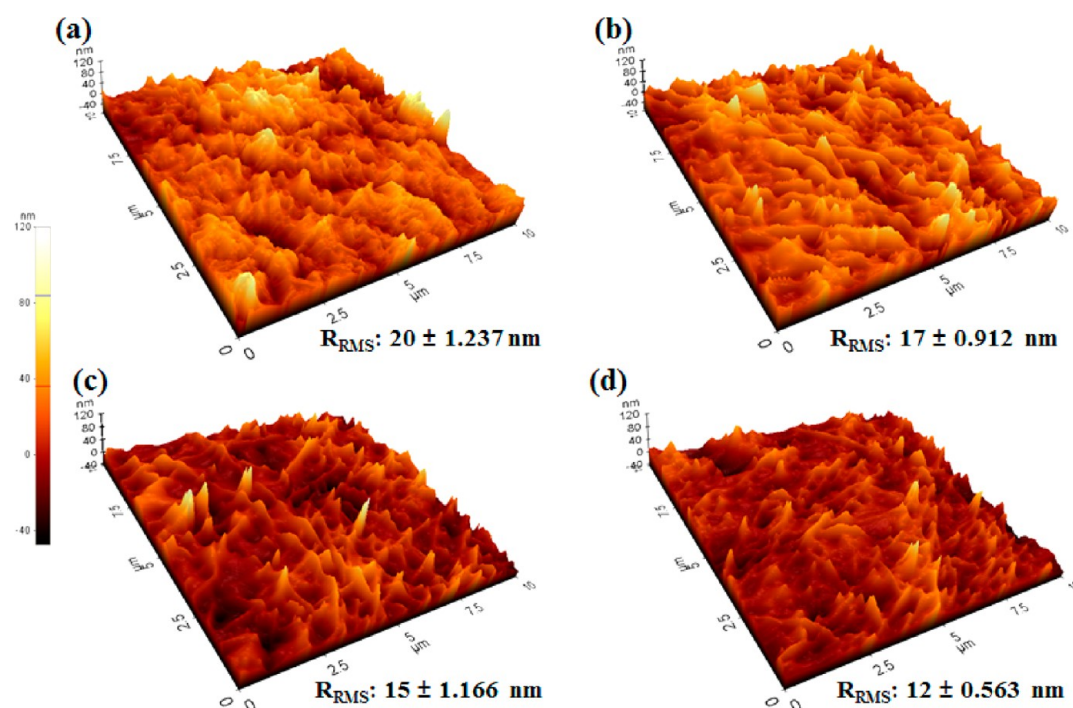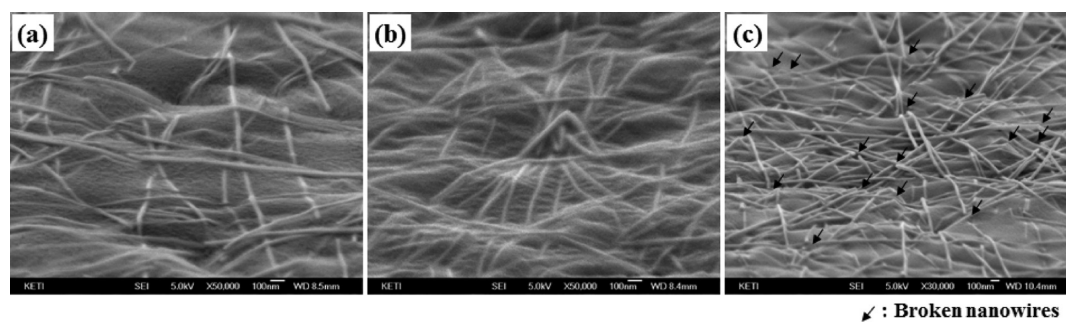
Figure 4. Effect of IPL irradiation on the morphology of AgNWs transferred to the surface of PUU: (a) as-transferred AgNWs; (b) after a single pulse of IPL irradiation; (c) after 4 pulses of irradiation; (d) after 8 pulses of irradiation.

↙ : **Broken nanowires**

Figure 5. FESEM images of AgNW electrodes on PUU: (a) pristine AgNWs transferred to the surface of PUU; (b) after 10 cycles of 100% stretching and releasing for AgNW/PUU after 4 pulses of IPL irradiation; (c) after 10 cycles of 100% stretching and releasing for AgNW/PUU without IPL irradiation (the disconnected wires are identified by arrows in the figure).

interaction between the nanowires and PUU. An interesting point is that the roughness of the AgNW film was decreased by the IPL irradiation. A comparison of Figure 4a−d shows that the root-mean-square roughness (R_{RMS}) of the as-transferred AgNW layer continuously decreased with repeated irradiation (see also the results from the measurements of larger areas in Figures S4 and S5 of the Supporting Information). For example, the value of R_{RMS} was reduced from 20 to 12 nm by 8 pulses of IPL irradiation. The microstructural evolution of the AgNW structure during a pulse of IPL irradiation can be divided into three stages.[26] First, highly intensive photonic energy was delivered to the AgNWs within a few hundreds or thousands of microseconds, thus instantly increasing the temperature to the melting point of Ag because of the photothermal effect.[27] Second, plasmonic welding of the AgNWs and removal of the PVP capping layer from their surface occurred as the temperature increased. Finally, the absorption of heat by irradiated AgNWs increased the temperature of AgNWs nearby, and the polymer underneath the nanowires was activated to react with the surface of

AgNWs. We determined that a further pulse of IPL irradiation would have intensified the fusion of the polymer to cause partial embedment of AgNWs in the surface of the polymer.[26] Once the AgNWs were partially buried at the polymer surface, it was difficult to remove them from the polymer. Because the underlying polymer could be melted by the "heat," it must be a thermoplastic material like PUU. As indicated in Figure 4, the surface of AgNW/PUU was planarized by the IPL irradiation possibly owing to the local fusion of PUU to the surface of nanowires. It was thus anticipated that the contact area between AgNWs and PUU was enlarged and the adhesion was enhanced. At the same time, R_s decreased by about 15% mainly as a result of the removal of PVP and plasmonic welding of the nanowires. However, whether the AgNWs were still exposed to air awaited investigation because many applications such as touch sensors or organic solar cells require sufficiently exposed electrodes for interconnection or carrier transference. If the AgNWs were fully embedded within the polymer matrix, conduction to the upper layer would have been prohibited, which would have resulted in very poor injection property, for
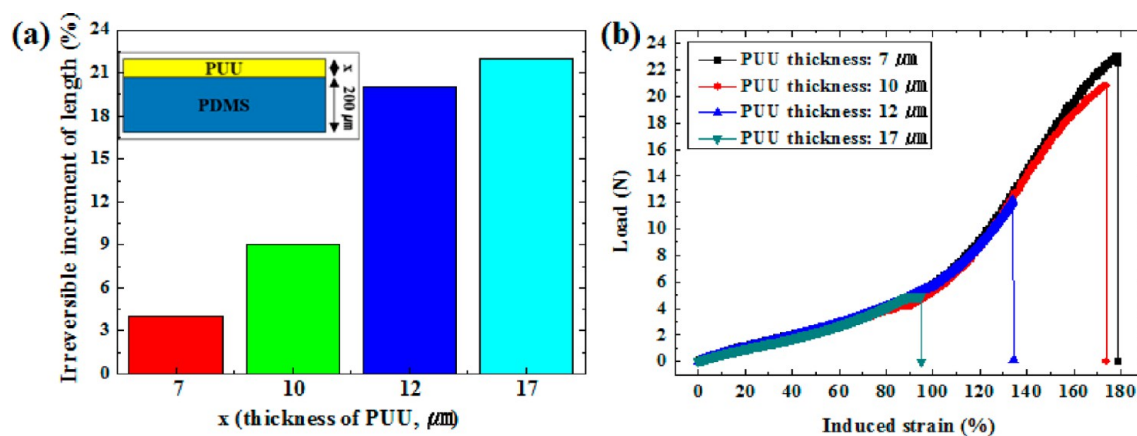
Figure 6. Effect of PUU thickness on (a) irreversible increment of length and (b) induced-strain−load curves for the PUU/PDMS structure.
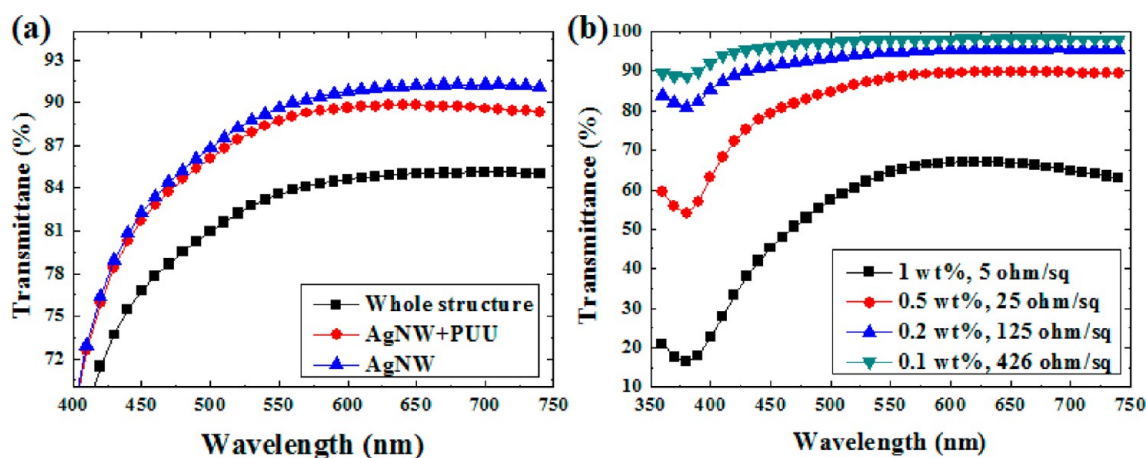

Figure 7. Transmittance of the AgNW/PUU/PDMS film: (a) comparison of transmittance for AgNW/PUU/PDMS, AgNW/PUU, and AgNW layers (R_s = 30 ohm/sq); (b) effect of nanowire density on transmittance of AgNW layer.

example. To examine whether the AgNWs were embedded or exposed, we immersed the fabricated samples in the Cu electroless plating solution for 10 min. The results (Figure S6 in the Supporting Information) showed that Cu was very densely plated onto the exposed AgNWs. Because the polymers were not physically or chemically affected by the immersion, it was determined that the AgNWs were sufficiently exposed to air.

After multiple rounds of IPL irradiation on the fabricated structure, we carried out the cyclic stretching test to evaluate the capability of the films to resist the repeated elongations. Figure 5a shows a tilt-view of the AgNW/PUU/PDMS layers (after 4 pulses of IPL irradiation) before the stretching test, whereas the same sample after 10 repeated cycles of 100% stretching−releasing is shown in Figure 5b. It was revealed in the figures that the nanowires were either partially or fully buried at the surface of PUU. The mechanism for this is discussed in an earlier section. After the stretching test, we could hardly find newly formed defects such as disconnections of wires or delamination of nanowire networks. As shown in Figure 5b, the wires were still buried in the polymer with high stability. When the IPL irradiation was eliminated, however, the mechanical stability was not sufficient to resist the stretching and many broken nanowires could be easily found, as shown in Figure 5c (top-view images for the damaged wires are also shown in Figure S7 of the Supporting Information). The instability of the percolated nanostructure originated from

insufficient adhesion between the constituents. For the AgNW/PUU structure, good adhesion between the AgNWs and PUU was the most important factor that prevented the nanowires from sliding at the interface. This sliding is known to be a main cause of the singularity in stress distribution, which contributes to the disconnection of stripped nanowires.

In addition, we also investigated the effect of PUU thickness (x) on the stretchability of the composite structure, focusing on the elasticity and breakup strain that can be induced. Because of the viscoelastic nature of PUU, the strain (50%) initially applied to PUU/PDMS was not reversibly recovered regardless of x, as shown in Figure 6a. The unrecovered length was defined as the extension when the load (stress) was equal to zero during the release of the stretched samples. The figure also shows that the lower the value of x, the smaller the irreversible strain in the film. When the PUU layer had the lowest value of x (7 μm), about 4% of the strain remained unrecovered. However, it should be noted that this value was not increased by further repetitions of the same amount of stretching. This is an important property because devices that are repeatedly stretched and released need to resist changes to their physical shape. In all cases, surface buckles were not formed after the stretching, as shown in Figure S8. We also investigated the induced-strain−load behavior with varying x. As shown in Figure 6b, when x was smaller than 10 μm, the capability to resist the breakup strain was nearly identical to that of the PDMS film alone. However, a further increase in x reduced the
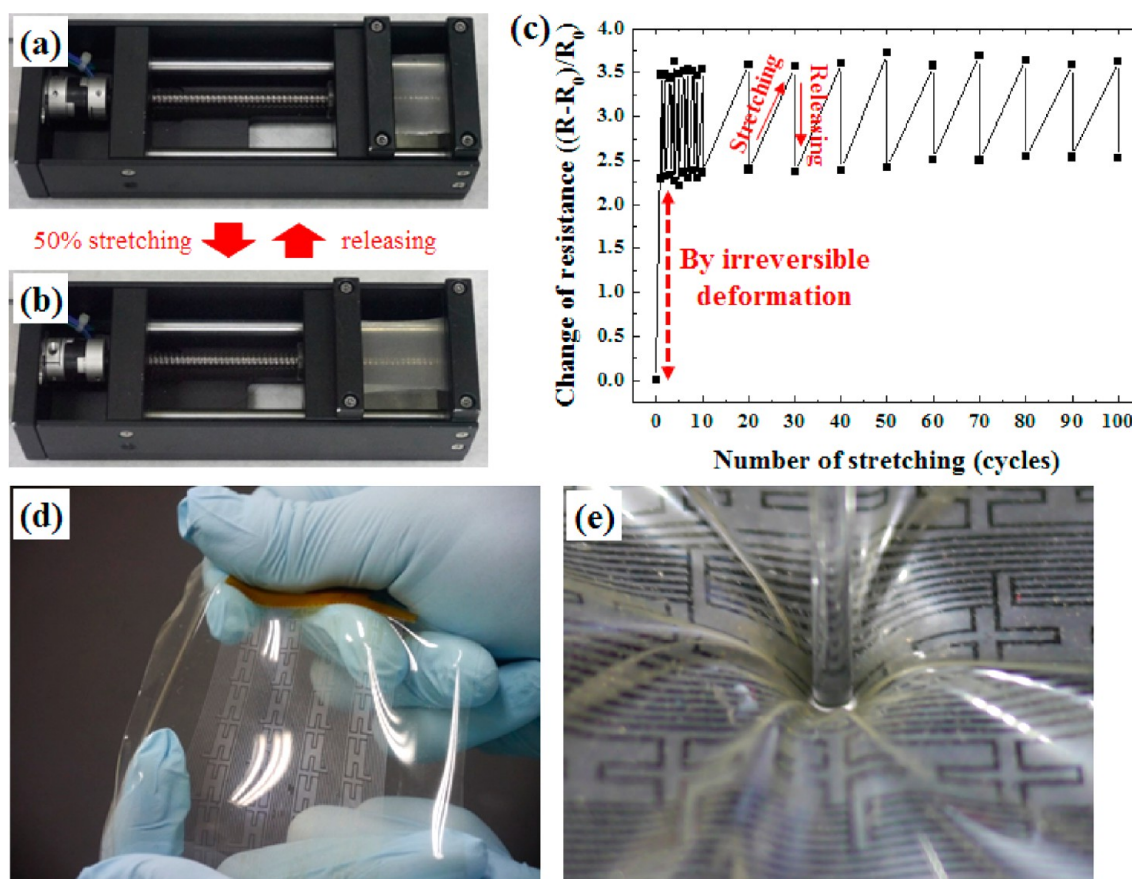
Figure 8. Stretching test of AgNW/PUU/PDMS films: (a) sample mounted on a stretching tester; (b) sample after 50% increase in film length; (c) effect of repeated stretching on the change in sheet resistance; (d) extension of the film; (e) stretched area created by pressing a film with the end of a glass rod.

resistance possibly owing to the thickness-dependent mechanical properties of cross-linked polymers.[28] Based on these results, we chose $x = 7$ μm as the lowest acceptable thickness for the PUU layer.

The light transmittance of the fabricated AgNW/PUU/PDMS film was measured in a visible range, as shown in Figure 7a. The concentration of the AgNWs in the coating dispersion was 0.4 wt %. The sheet resistance of the film was R_s = 30 ohm/sq, and the transmittance was measured with various reference media: air, PDMS, and PUU/PDMS. Using air as the reference medium, the transmittance of the entire structure was measured, while the reference media of PDMS and PUU/PDMS were used for measurements of the transmittance of AgNW/PUU and mere AgNWs, respectively. A comparison of the plots in Figure 7a shows that PUU was exceptionally transparent at wavelengths in the range of 400−500 nm. Therefore, it is beneficial to employ PUU together with yellowish materials like AgNWs or polyimide. The transmittance of the entire structure was significantly lower than those of AgNW/PUU and mere AgNWs owing to the high thickness of the PDMS employed (200 μm). The effect of AgNW density can be seen in Figure 7b, with R_s showing a significant decrease as the concentration of the AgNW dispersion was increased. This implies that the lateral conductance of the composite film was predominantly determined by the AgNW density. The combination of transmittance and R_s of the composite film is comparable to

those of sputtered crystalline ITO, and the sheet resistance could be reduced to <10 ohm/sq, the value for the ITO glass.

Given that the primary goal of developing AgNW/PUU/PDMS electrodes is to obtain highly reliable mechanical performance under continuous large-strain deformation, repeated stretch-and-release tests were carried out on the films fabricated as part of this study. An automated testing tool was employed, which enabled the electrode to experience alternate stretching and releasing repeatedly, resulting in cyclic fatigue failure. This test used an elongation of 50% and the cycle was repeated up to 100 times. Figure 8a,b shows the released and stretched state, respectively, of a sample mounted on a stretching module. Figure 8c shows the evolution of resistance of the transparent electrode (transmittance at 550 nm for AgNW/PUU: 88%; R_s: 25 ohm/sq) during the cyclic stretching−releasing test (the result of an in situ measurement is also shown in Figures S9 and S10 of the Supporting Information). The resistance of the AgNW network was sharply increased at the very first stretching, but it did not return to the starting value even after the strain was fully released. This irreversibility was mainly due to the inelastic deformation caused by the first stretching of the AgNW/PUU/PDMS electrode. Beginning with the second cycle, however, the resistance at the strain minima increased to 2.3 times the initial value, and reversibility was certainly observed. The reversibility continued until the stretching−releasing reached 100 cycles. The stability of the electrodes during stretching and releasing was also be confirmed by Movies S1 and S2 of the Supporting

Information. Another notable feature was that the increase in resistance by the stretching after the first cycle remained below 50%. To the best of our knowledge, this is one of the most stable stretchable transparent electrodes ever reported. Figure 8d,e shows the stretchability of the fabricated patterned electrodes with various deforming motions (see also Figure S11 of the Supporting Information). This high mechanical stability originated from the good adhesion between AgNWs and PUU, and the interfacial areas between them were enlarged by IPL irradiation. Only one issue remains unresolved: the inelastic deformation formed during the first stretching. From a practical perspective, this can be settled by a simple prestraining before the material is used; the development of a fully elastic polymer for this structure is the subject of our future studies.

CONCLUSION

A highly stretchable, transparent, and conductive electrode was fabricated using a combination of percolated AgNWs, PUU, and PDMS by employing an inverted pattern transfer method. AgNW electrodes fabricated on a Kapton film were perfectly transferred to the surface of PUU, a polymer specially designed to have a high adhesion to AgNWs by the incorporation of 2,2-bis(hydroxymethyl)butyric acid with a carboxylic group. The sheet resistance of the AgNW networks was not severely elevated by the transfer, and the transferred nanowires were stable with moderate surface roughness. Multiple exposures to IPL irradiation led to partial melting of PUU, which resulted in embedment of AgNWs in the PUU and enhancement of the mechanical stability. By optimizing the thickness of PUU on the PDMS layer, irreversible deformation could be minimized. The measured transmittance and sheet resistance of the electrode revealed that the properties of the films were comparable to those of ITO-based electrodes. A cyclic stretching test revealed that the developed electrodes were highly stretchable with exceptional stability. The development of the material and the fabrication method presented here are expected to offer guidelines for the fabrication of stretchable electronic devices.

ASSOCIATED CONTENT

ⓢ Supporting Information

Figures and movies of additional experimental data. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsami.5b04693.

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Author Contributions
The paper was written through contributions of all authors. J.-W. Kim designed and supervised the research. J.-W. Kim and Y. Kim wrote the paper. D.-H. Kim and K.-C. Yu participated in the fabrication of the composite electrode and evaluation. All authors have given approval to the final version of the paper.

Notes
The authors declare no competing financial interest.

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ABBREVIATIONS

AgNW, silver nanowire
IPL, intense pulsed light
ITO, indium−tin oxide
PDMS, poly(dimethylsiloxane)
PU, polyurethane
PUU, polyurethane−urea
PVP, poly(vinylpyrrolidinone)
x, PUU layer thickness
R_s, sheet resistance

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