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Highly Conductive and Flexible Silver Nanowire-Based Microelectrodes on Biocompatible Hydrogel

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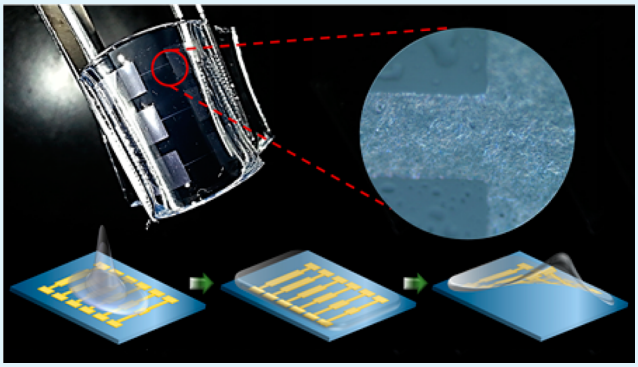
Supporting Information

ABSTRACT: We successfully fabricated silver nanowire (AgNW)-based microelectrodes on various substrates such as a glass and polydimethylsiloxane by using a photolithographic process for the first time. The AgNW-based microelectrodes exhibited excellent electrical conductivity and mechanical flexibility. We also demonstrated the direct transfer process of AgNW-based microelectrodes from a glass to a biocompatible polyacrylamide-based hydrogel. The AgNW-based microelectrodes on the biocompatible hydrogel showed excellent electrical performance. Furthermore, they showed great mechanical flexibility as well as superior stability under wet conditions. We anticipate that the AgNW-based microelectrodes on biocompatible hydrogel substrates can be a promising platform for realization of practical bioelectronics devices.



KEYWORDS: *silver nanowire, hydrogel, biocompatible electrode, bioelectronic device*

The bioelectronic devices have been studied and progressed for decades to offer improved healthcare and environmental protection.[1−4] Because biological tissues have a dynamic and curvilinear biological environment, it is necessary to fabricate flexible bioelectronic devices on biocompatible substrates. Recently, many research groups have demonstrated the great progress on the bioelectronic devices based on microelectrode arrays onto polymeric hydrogels.[5−8] Polymeric hydrogels are promising materials for the bioelectronic devices that are contacted with biological environment because of their hydrophilic nature, biocompatibility, and similar mechanical properties with human soft tissue.[9−12] For fabrication of the bioelectronic devices based on polymeric hydrogels, it is necessary to develop flexible microelectrodes. However, there have been rare reports on the hydrogel-based flexible microelectrodes for bioelectronic devices because of the difficult fabrication of patterned microelectrodes.[13] The mechanical flexibility of the microelectrodes is of prime importance in bioelectronic devices because they have to conform to the curvilinear shapes of biological tissues and form excellent electrical contacts.[14−17] Therefore, flexible, biocompatible, and highly conductive microelectrodes have been required for the next-generation bioelectronic devices. Recently, carbon nanotube, conducting polymers, and graphene on the hydrogel substrates have been reported as the flexible and biocompatible microelectrodes for bioelectronic applications.[18−25] For example, the microelectrodes based on carbon nanotube or conducting polymer were shown to reduce electrical impedance at the interface with various proteins and tissues in physiological conditions.[26,27] However, compared to metal electrodes such as gold, platinum, silver, and iridium, the relatively low electrical conductivity of the carbon nanotube and conducting polymers have limits on its practicality for bioelectronic applications.[28,29] Therefore, it is essential to develop new flexible conducting materials for practical bioelectronic devices.

Recently, one-dimensional silver nanowire (AgNW) has received a great attention as a novel material for a flexible transparent conducting electrode (TCE) because of its remarkable physical properties such as the excellent electrical and optical properties, and mechanical robustness.[30−34] The AgNW TCE can be prepared at a low processing temperature by solution processes such as spin-coating and bar-coating. In addition, a percolated network of the AgNW could be coated onto rough and flexible substrates.[35−37] Therefore, compared to the other solution-processed conductive materials such as metal nanoparticles, carbon nanotubes, and conducting polymers, which possess lower electrical conductivity and poor flexibility, the AgNW is in a better position to the fabrication of flexible microelectrodes for the bioelectronic devices.[38−41] For the successful application of the AgNW-based microelectrodes to the bioelectronic devices, it is strongly required to develop the micropatterning technique for the AgNW onto the hydrogel substrate. Moreover, the fabrication of the AgNW-based

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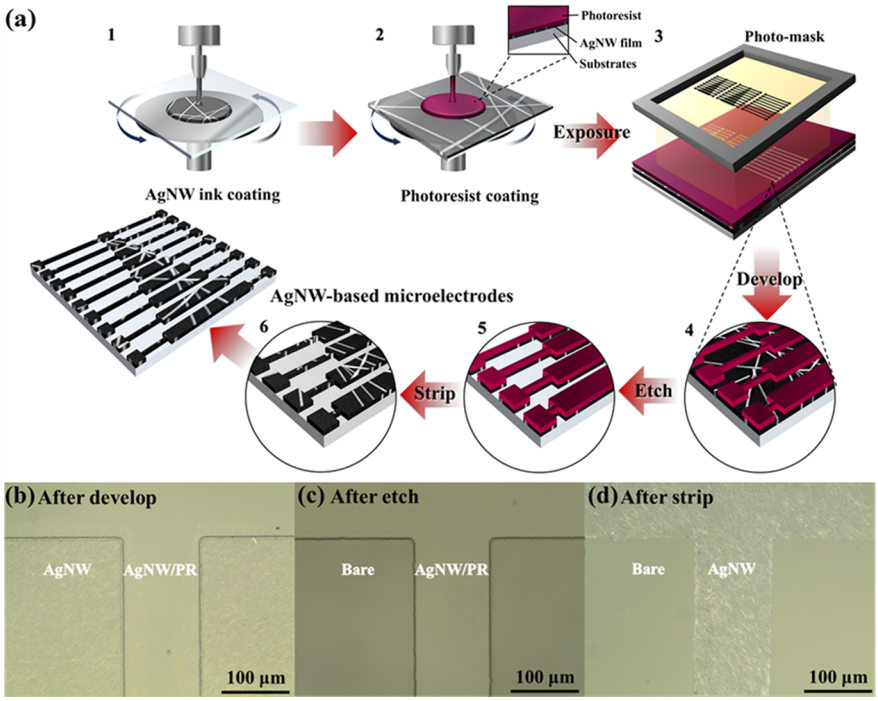

Figure 1. (a) Schematic illustration for fabrication of AgNW-based microelectrodes using a photolithographic process. The optical microscope (OM) images of AgNW micropatterns (b) after developing the PR, (c) after etching the AgNW, and (d) after stripping the PR.

microelectrodes on various substrates is desirable to extend its applicability in extensive electronics fields. Consequently, there have been a need for developing a new patterning technique of AgNW-based microelectrodes onto various substrates without loss of electrical and mechanical properties.

In this study, we successfully fabricated highly conductive AgNW-based microelectrodes on various substrates such as a glass or polydimethylsiloxane (PDMS) by using a photolithographic process. Moreover, we developed the patterning technique of the AgNW-based microelectrodes on a biocompatible hydrogel substrate through direct transfer process. The AgNW-based microelectrodes on the hydrogel exhibited high electrical conductivity and excellent mechanical flexibility. We anticipate that the AgNW-based microelectrodes on the hydrogel can be a promising platform for realization of practical bioelectronics devices.

The AgNW, which had an average diameter and length of 20−40 nm and 10−20 μm, respectively, was used in this study. The percolated network of the AgNW was able to provide continuous electron transport pathways, leading to excellent electrical properties. Moreover, the AgNW film showed strong mechanical adhesion on a glass substrate. We carried out a cross-cut test with a 3 M scotch tape for assessment of the adhesion property of the AgNW film. None of the square lattice of the AgNW film was detached, verifying that the AgNW film possessed strong adhesion property, which is essential for patterning technique such as a photolithography. Therefore, we performed a photolithographic process for the AgNW film to obtain various pattern sizes of AgNW-based microelectrodes. As illustrated in Figure 1, the AgNW-based microelectrodes with a variety of pattern width from 20 to 200 μm were successfully prepared on a glass substrate by using a photolithographic process.

The SEM images in Figure 2a−c and the three-dimensional Nano View image in Figure S2 in the Supporting Information

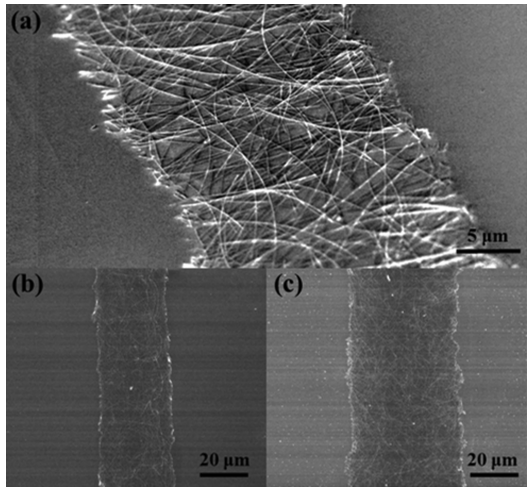

Figure 2. SEM images of AgNW-based microelectrodes with different pattern width (a) 20, (b) 30, and (c) 40 μm.

clearly display that the AgNW micropatterns with various width from 20 to 200 μm on a glass substrate were successfully fabricated. To open up the possibilities of practical applications of the AgNW-based microelectrodes, we also tested the micropatterning of the AgNW on a polymer substrate such as a polydimethylsiloxane (PDMS) which has hydrophobic surface and very high surface roughness. The AgNW micropatterning based on the photolithographic process was also successfully achieved on the PDMS substrate (see Figure S3 in the Supporting Information). It is noteworthy that this is the first report on the AgNW-based microelectrodes on the PDMS substrate achieved by the photolithographic patterning process.

To demonstrate the electrical property of the AgNW-based microelectrodes, we prepared a AgNW film on a glass substrate by spin-coating a AgNW ink at 300 rpm. The sheet resistance

Table 1. Resistances of AgNW-Based Microelectrodes with Different Width from 20 to 200 μm and Same Length (5 mm)

width (μm)	20	40	60	80	100	150	200
resistance (Ω)	3349 ± 149	1784 ± 56	1219 ± 56	968 ± 25	740 ± 56	548 ± 49	479 ± 34

Table 2. Resistances of AgNW-Based Microelectrodes with Different Spin-Coating Speed and Same Dimension (100 μm in width and 5 mm in length)

spin-coating speed (rpm)	300	400	500	600	700
resistance (Ω)	740 ± 56	1283 ± 66	1667 ± 76	2751 ± 106	4007 ± 248

of the AgNW film was 21.7 Ω/sq. Then, we fabricated the AgNW-based microelectrodes which have the same pattern length (5 mm) and different pattern width from 20 to 200 μm. As shown in Figure 3a, the AgNW-based microelectrodes

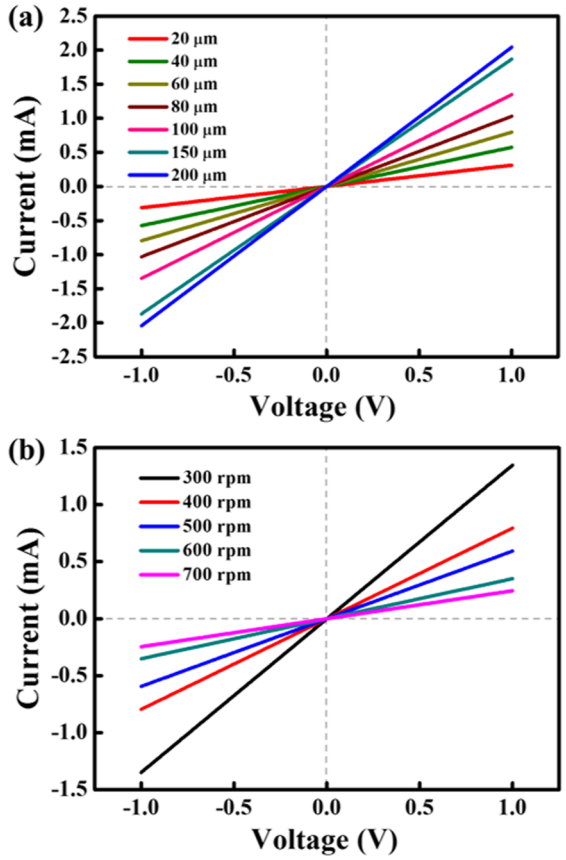

Figure 3. Current–voltage (I–V) characteristics of AgNW-based microelectrodes with (a) the different width from 20 to 200 μm and 5 mm in length and with (b) the different spin coating speed from 300 to 700 rpm and same dimension (100 μm in width and 5 mm in length).

showed linear I–V characteristics, indicating the characteristic of metallic conductors. The current–voltage (I–V) characteristics of the AgNW-based microelectrodes were measured by a two-point probe station. The contact resistance between the probe tips and the AgNW-based microelectrodes were tested before measuring the electrical resistance of the AgNW-based microelectrodes. It exhibited much lower resistance (~1.5 Ω) than the electrical resistance of the AgNW-based microelectrodes. The average electrical resistance of the AgNW-based microelectrodes was obtained from over 20 samples. The electrical resistance of the AgNW-based microelectrodes decreased from 3.35 KΩ to 479 Ω as the pattern width of the AgNW-based microelectrodes increased from 20 to 200 μm. These results clearly demonstrate that the electrical conductivity of the AgNW-based microelectrodes depends on the pattern width.

To investigate the effect of the AgNW network density on the electrical conductivity, spin-coating speed for preparation of the AgNW films was varied from 300 to 700 rpm. As shown in Figure S4 in the Supporting Information, the AgNW network density was decreased as spin-coating speed increased. When the spin-coating speed increased from 300 to 700 rpm, the sheet resistance of the AgNW films increased from 21.7 to 91.3 Ω/sq. Figure 3b shows the relationship between the electrical conductivity and the AgNW network density of the AgNW-based microelectrodes. We measured I–V characteristics of various AgNW-based microelectrodes with different AgNW network density. The pattern length and width of all the AgNW-based microelectrodes were 5 mm and 100 μm, respectively. The electrical conductivity of the AgNW-based microelectrodes decreased as the spin-coating speed increased. The AgNW-based microelectrode prepared at a spin speed of 300 rpm showed the lowest resistance of 740 Ω, whereas the AgNW-based microelectrode prepared at a spin speed of 700 rpm exhibited the highest resistance of 4.01 kΩ. The increase of spin-coating speed gave rise to a decrease in the electrical conductivity of the AgNW-based microelectrodes because the sparse network of the fewer AgNW resulted in the reduced electron transport pathways. We also measured the impedance of the AgNW films prepared at different spin-coating speeds in a physiological saline solution. The impedance of the AgNW film with surface area of 1 × 1 cm^2 was measured in the frequency range from 10 to 1000 Hz (see Figure S5 in the Supporting Information). The impedance measurements of the AgNW-based microelectrodes as a function of spin-coating speed also showed similar results like the electrical conductivity test. These results verify that the electrical conductivity of the AgNW-based microelectrodes can be easily controlled by changing the AgNW network density.

To develop flexible and biocompatible microelectrodes based on the AgNW for bioelectronic applications, we conducted experiments to fabricate the AgNW-based microelectrodes onto hydrogel substrates. Hydrogels are polymeric materials that are capable of absorbing water without degradation of three-dimensional structure.[42−45] We have chosen a hydrogel based on polyacrylamide (PAM) as a substrate for the fabrication of the AgNW-based biocompatible microelectrodes. PAM has been extensively utilized in various applications as the biomaterial because of inert, biodegradable, hydrophilic, and biocompatible properties.[45−50] PAM-based cross-linked hydrogel was possibly synthesized by polymerization of acrylamide monomer with N,N′-methylenebis(acrylamide) (MBA), ammonium persulfate (APS), and N,N,N,N′-tetramethylethylenediamine (TMEDA) to act as a cross-linker, an initiator, and an accelerator, respectively. The concentration of each reagent

could have an effect on the polymerization reaction. The concentrations of acrylamide monomer and cross-linker MBA could affect the swelling capacity of the hydrogel.[42,46] High concentration of acrylamide monomer and MBA tended to decrease the water absorbency due to higher cross-linking density. Lower water absorbency could not possess the structural stability and flexibility.[51] The concentrations of initiator APS and accelerator TMEDA affected the polymerization time of the hydrogel. The polymerization time of the hydrogel could be reduced by increasing the concentrations of APS and TMEDA.[52,53] Therefore, we tested various combinations of reagents for synthesis of the hydrogels. The proper amount of each reagent was determined to control the polymerization reaction (acrylamide:MBA:APS:TMEDA = 1:0.023:0.016:0.006).

We fabricated the AgNW-based microelectrodes onto the PAM-based cross-linked hydrogel by using the photolithographic process as described above. However, although the AgNW ink was successfully coated on the hydrogel, the annealing process to dry the AgNW ink and PR damaged the three-dimensional structure of the moist hydrogel substrate. Therefore, we developed a direct-transfer process of the AgNW-based microelectrodes from a glass to a hydrogel during the hydrogel polymerization. The direct transfer process of the AgNW-based microelectrodes during the hydrogel polymerization allows the AgNW micropattern to be monolithically embedded at the surface of the hydrogel. The fabrication process of the AgNW-based microelectrodes on the hydrogel substrate is schematically illustrated in Figure 4a. First, the AgNW-based microelectrodes on a glass substrate were prepared by the photolithographic process. After the fabrication of the AgNW-based microelectrodes on the glass substrate, the prepolymer solution for the hydrogel was prepared, and then immediately poured over the AgNW-based microelectrodes on the glass substrate. When the prepolymer solution was cast onto the AgNW-based microelectrodes, it penetrated into the percolated network of the AgNW-based microelectrodes because of the low viscosity of the prepolymer solution. When the hydrogels built the polymer networks, polymer chains in the hydrogel were cross-linked with the AgNW-based microelectrodes. The strong adhesion between the AgNW-based microelectrodes and the hydrogel led to a successful transfer of the AgNW-based microelectrodes from the glass substrate to the hydrogel.

The degree of polymerization could have an effect on the transfer of the AgNW-based microelectrodes from the glass to the hydrogel. If the polymerization was not completely finished, the AgNW-based microelectrodes were partially transferred onto the hydrogel. Therefore, before peeling the hydrogel, acrylamide monomers in prepolymer solution had to be completely polymerized for the perfect transfer of the AgNW-based microelectrodes. It was found that at least 20 min of reaction time was required for complete polymerization of the acrylamide monomers. It is noteworthy that the AgNW-based microelectrodes could be nondestructively transferred in 20 min. Finally, the hydrogel was peeled off the glass substrate to obtain the AgNW-based microelectrodes on the hydrogel. Figure 4b, c show the images of the AgNW-based microelectrodes on the glass substrate. Figure 4d, e display the images of the AgNW-based microelectrodes on the hydrogel after the direct transfer process. These results clearly confirm that the AgNW-based microelectrodes can be completely

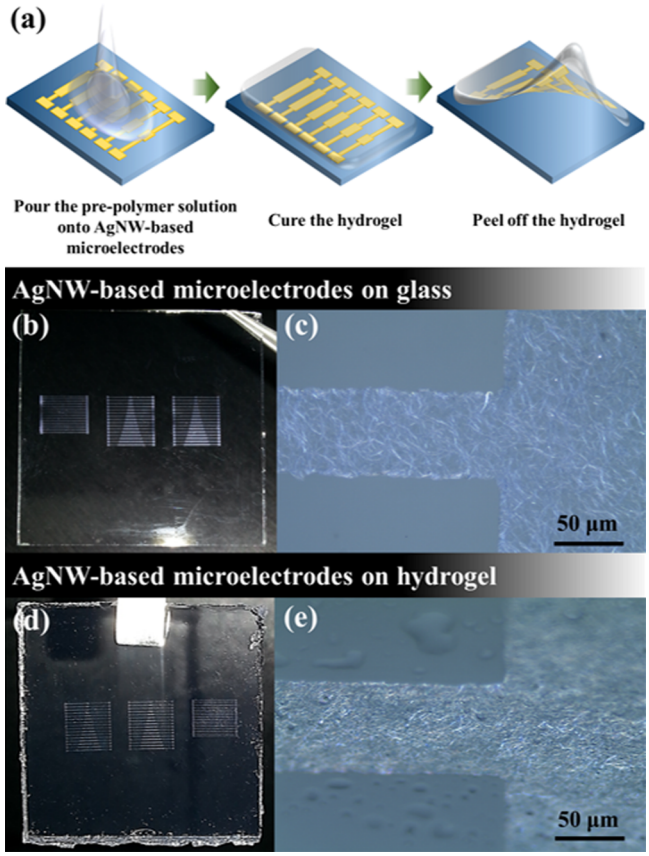

Figure 4. (a) Schematic illustration of the transfer of AgNW-based microelectrodes to hydrogel substrate. (b) Photograph and (c) OM image of AgNW-based microelectrodes on the glass before transfer. (d) Photograph and (e) OM image of AgNW-based microelectrodes on the hydrogel.

transferred from the glass to the hydrogel without any structural damages.

To investigate the electrical property of the AgNW-based microelectrodes on hydrogel, the $I-V$ characteristic of AgNW-based microelectrode with 200 μm in width and 5 mm in length was measured by using a probe station. Figure 5a shows the $I-V$ characteristics of the AgNW-based microelectrodes on the glass and hydrogel substrates before and after the transfer process. The $I-V$ characteristics of the AgNW-based microelectrodes on both substrates were measured to evaluate the change in the electrical property of the AgNW-based microelectrode after the transfer to hydrogel. The AgNW-based microelectrodes on both substrates exhibited the linear $I-V$ behaviors. The electrical resistance of the AgNW-based microelectrodes on the glass was 103 Ω. The electrical resistance of the AgNW-based microelectrodes on the hydrogel was 109 Ω. Compared to the resistances of the AgNW-based microelectrode on the glass, there is no significant change in the AgNW-based microelectrodes on the hydrogel. This result apparently indicates that the electrical properties of the AgNW-based microelectrodes have not been deteriorated during the direct transfer process.

The mechanical flexibility of the microelectrodes is of prime importance in bioelectronic devices because biological tissues have dynamic and three-dimensional structures.[15,54,55] To demonstrate the mechanical flexibility of the AgNW-based microelectrodes, we performed the bending test. In this test,

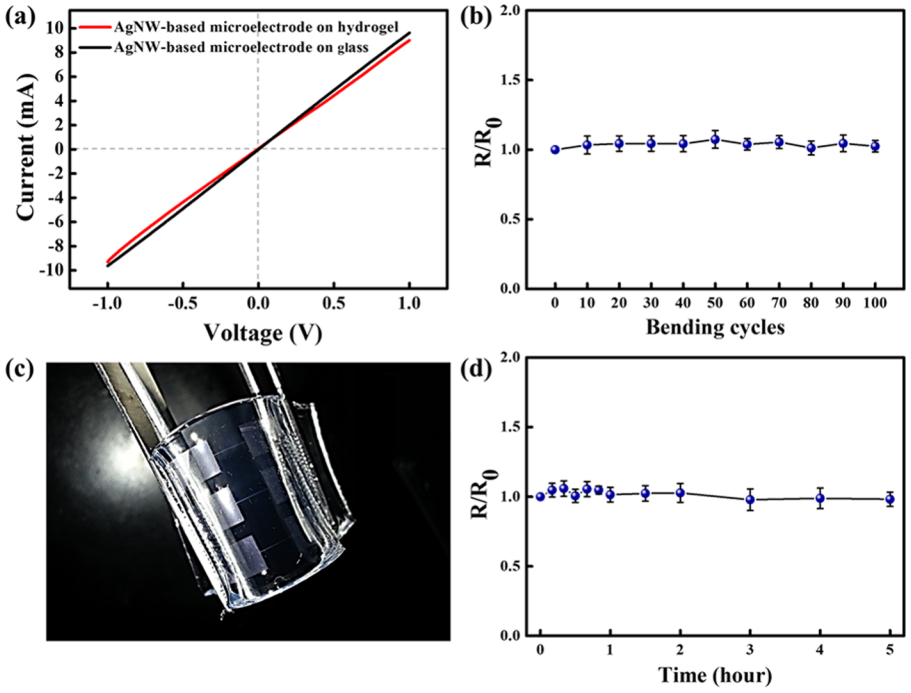

Figure 5. (a) Current−voltage (I−V) characteristics of AgNW-based microelectrodes on hydrogel and AgNW-based microelectrodes on glass which have 200 μm in width. (b) Flexibility test of AgNW-based microelectrodes on hydrogel. (c) Photograph of bended AgNW-based microelectrodes on hydrogel. (d) Stability test of AgNW-based microelectrodes on hydrogel in physiological saline solutions.

the AgNW-based microelectrodes on hydrogel were rolled around a bending radius of 5 mm, subsequently unrolled, and repeated for 100 bending cycles. The electrical resistance of the AgNW-based microelectrodes was measured in every tenth bending cycle, and the electrical resistances were compared to initial value. As shown in Figure 5b, there was no significant change in the resistance of the AgNW-based microelectrode for 100 bending cycles. In addition, there was no buckling and fracture of the AgNW after the bending test (Figure 5c). These results clearly confirm that the AgNW-based microelectrodes onto the hydrogel substrate are mechanically stable because of the excellent flexibility of the AgNW and hydrogel.

To evaluate the stability of the AgNW-based microelectrodes in the wet condition, we immersed the AgNW-based microelectrodes on the hydrogel in DI water. The change in the electrical resistance of the AgNW-based microelectrodes was measured and compared to initial value. The AgNW-based microelectrodes were completely immersed in a physiological saline solution for 5 h. The hydrogel was swollen by about 10% after the stability test. However, as shown in Figure 5d, the electrical resistance of the AgNW-based microelectrodes remained unchanged, verifying that the AgNW-based microelectrodes on hydrogel possess the superior stability in the physiological condition. This result means that the AgNW-based microelectrodes on hydrogel could be applied in various bioelectronic devices, which are used in physiological conditions.

In conclusion, we successfully fabricated the AgNW-based microelectrodes on glass and plastic substrates using a photolithographic process for the first time. The AgNW-based microelectrodes exhibited excellent electrical properties and mechanical flexibility. The electrical conductivity of AgNW-based microelectrodes could be controlled by pattern dimension and network density of the AgNW. We also demonstrated the optimized direct transfer process of AgNW-based microelectrodes from a glass to a biocompatible PAM-based hydrogel. The AgNW-based microelectrodes were successfully fabricated on the biocompatible hydrogel with cross-linking between polyacrylamide and AgNW-based microelectrodes. The AgNW-based microelectrodes on the biocompatible hydrogel showed excellent electrical performance. Furthermore, they showed great mechanical flexibility as well as the superior stability under wet conditions. We anticipate that the AgNW-based microelectrodes on biocompatible hydrogel substrates can act as a promising platform for realization of practical bioelectronics devices.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The preparation and characterization of AgNW-based microelectrodes with various conditions, and detailed information about materials are included. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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