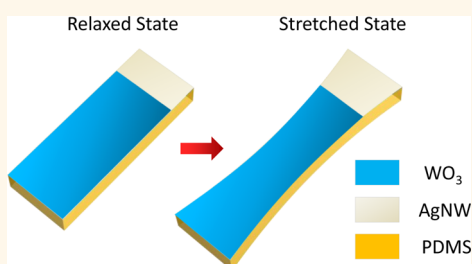


Stretchable and Wearable Electrochromic Devices

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ABSTRACT Stretchable and wearable WO₃ electrochromic devices on silver nanowire (AgNW) elastic conductors are reported. The stretchable devices are mechanically robust and can be stretched, twisted, folded, and crumpled without performance failure. Fast coloration (1 s) and bleaching (4 s) time and good cyclic stability (81% retention after 100 cycles) were achieved at relaxed state. Proper functioning at stretched state (50% strain) was also demonstrated. The electrochromic devices were successfully implanted onto textile substrates for potential wearable applications. As most existing electrochromic devices are based on rigid technologies, the innovative devices in their soft form hold the promise for next-generation electronics such as stretchable, wearable, and implantable display applications.



KEYWORDS: stretchable electronics · wearable electronics · electrochromic · elastic conductors · silver nanowires · flexible displays

Electrochromism is a type of nonemissive technology that can controllably tune optical properties by electrical signals.^{1–3} They have found important applications in the fields of electronic paper-like displays (e-paper), antiglare rear-view mirrors in cars, energy-saving smart windows in buildings, active camouflage, to name a few.^{4,5} Most previous electrochromic devices were fabricated on rigid substrates, typically transparent conductive glass substrates.¹ However, the compelling needs of next-generation electronics would require flexible and even stretchable electrochromic devices.^{4,5} For example, flexible displays are required for the fabrication of e-papers and e-books that can possibly be folded like a regular paper. For more sophisticated and futuristic needs, the electrochromic displays are potentially stretchable and wearable for future smart clothes and implantable display applications. Imparting stretchability to conventionally rigid electrochromic devices is technically much more challenging than achieving flexibility. The soft, stretchable form of electrochromic devices is mechanically robust and can be stretched, twisted, folded, and crumpled without performance degradations. They can conform to complex nonplanar surfaces and enable unprecedented applications that are unreachable with existing rigid technologies.

Polymers and metal oxides are two types of dominant electrochromic materials. Polymers are considered as promising candidates for stretchable applications due to their intrinsically soft molecular chains.^{6–9} The rigid inorganic metal oxides such as WO₃, NiO, TiO₂, and MoO₃, on the contrary, may crack and degrade upon stretching, although oxide materials are desired electrochromic materials due to their higher coloration efficiency (CE) and better cyclic and environmental stability.^{10–12} It would be of interest to design stretchable devices that incorporate inorganic oxide materials with superior electrochromic properties. Existing electrode materials such as the most widely used indium tin oxide (ITO) also encounter severe stretchability challenges. In this report, we demonstrate the fabrication of highly stretchable WO₃ electrochromic devices based on embedded elastic conductors.

Among the reported methods for stretchable device fabrications, designing structures that stretch is the most successful strategy to impart stretchability to originally rigid materials.¹³ For example, wavy and arc-shaped nanostructures were designed to effectively accommodate strains from various forms of deformations.^{14,15} Percolating nanowire (NW) networks were found to show fairly good tolerance to stretching

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before they rupture into patches through the inter-sliding of entangled NWs.^{16–21} Herein we employ AgNW networks embedded in polydimethylsiloxane (PDMS) elastomer matrix as stretchable conductors for

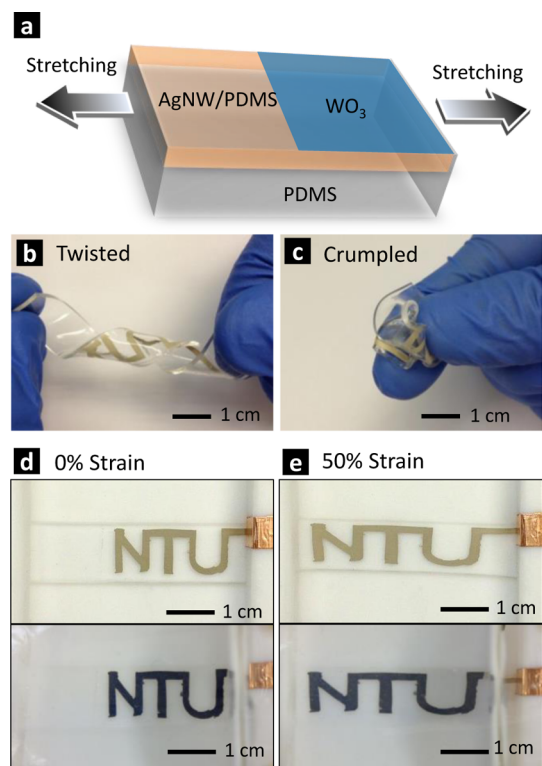


Figure 1. (a) Schematic of the stretchable electrochromic device. (b,c) Stretchable devices being twisted and crumpled, showing excellent mechanical robustness. (d,e) Examples of the patterned device in bleached and colored states at 0 and 50% strain, respectively.

electrochromic devices. Electrochromic WO₃ layer was fabricated *via* electrochemical deposition and exhibited excellent stretchability owing to the noncontinuous morphologies following the exposed AgNWs on the top surface. The WO₃ electrochromic electrodes were also implanted on cotton textile substrates, demonstrating their potential applications in wearable electronics.

RESULTS AND DISCUSSION

Representative examples of the stretchable electrochromic devices are shown in Figure 1. The device is based on AgNW/PDMS elastic conductor with electrochemically deposited WO₃ active layer (Figure 1a). The elastic conductors were fabricated *via* a lithographic filtration method²⁰ which enables the fabrication of stretchable electrodes with desired patterns. Instead of those electrodes with conducting paths such as CNTs on top of the elastomer substrates,¹⁸ the AgNWs were embedded inside the elastomer matrix with the top-layer NWs exposed for electrical contact (Figure 2). The unique embedded structures possess excellent stretchability, significantly improved durability against mechanical scratching, and are free of delamination and peeling off problems upon repeated stretching. The as-fabricated stretchable electrochromic devices are mechanically robust and can be stretched, twisted, folded, and crumpled without performance degradations (Figure 1b–e; see also Figure S1, Supporting Information), making them very attractive candidates for next-generation stretchable and wearable electrochromic display applications. Typical images of the colored and bleached stretchable devices in relaxed and stretched (50% strain) states are shown in

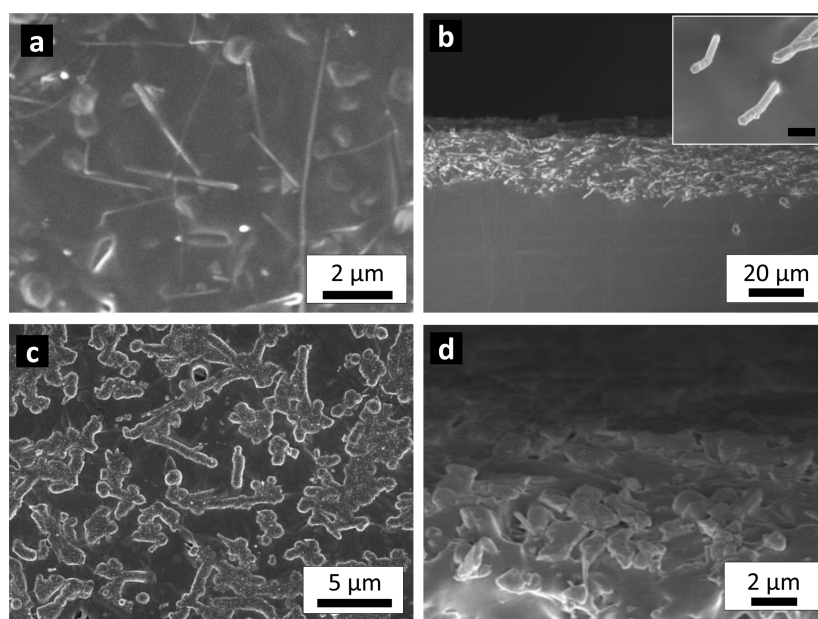


Figure 2. Top and cross-sectional views of the stretchable AgNW electrodes (a,b) before and (c,d) after WO₃ deposition. Inset in (b) is an enlarged cross-sectional view showing the exposed NW tips from the elastomer matrix. Scale bar: 500 nm.

Figure 1d,e, respectively. It is evident that the devices maintained their electrochromic properties even in highly stretched states.

Detailed structural characterizations of the stretchable devices are shown in Figure 2 and Figure S2, Supporting Information. Top view (Figure 2a) and cross-sectional view (Figure 2b) scanning electron microscopy (SEM) images confirm that the AgNWs are embedded in the elastomer matrix with only the top NW layer exposed as conducting sites for electrochemical deposition. Inset in Figure 2b is an enlarged view of the freshly cut cross section showing the exposed NW tips from the surrounding PDMS matrix. WO_3 , the most widely studied electrochromic oxide with high contrast, high CE, long-term cyclic stability, and ideal transparent/blue color switching,^{2,12} was used for our prototype demonstration. WO_3 layer was deposited at a constant voltage of -0.5 V (versus Ag/AgCl) in a three-electrode system. The deposited WO_3 film followed the arrangements of exposed NW surfaces (Figure 2c) since the rest of the top surfaces are covered with insulating PDMS. We suggest that the discretely distributed WO_3 microstructures are beneficial for stretchable applications. The microstructures have firm contact with underlying AgNWs since they were deposited directly on AgNWs. Upon stretching, WO_3 -coated AgNWs tend to rotate and orient toward the stretching direction.^{18,22} However, since the strain was accommodated by the intersliding and rearrangements of AgNWs and not the mechanical elongation of individual AgNW, the deposited WO_3 microstructures can maintain their integrities in stretched states. The noncontinuous morphology also ensures that they are more resistant to stretching as compared to

continuous films which may crack into microscale patches upon mechanical elongation of the entire film.²³

Electrochromic properties of the stretchable devices at relaxed state are shown in Figure 3. The as-deposited WO_3 active layer was shown to be amorphous^{24,25} and can change its color from transparent to blue upon cation (such as H^+ , Li^+) intercalation. The coloration and bleaching (charge/discharge) processes in acidic environments are based on the following reversible reactions:^{26,27}



Electrochromic properties were characterized in reflective mode using a two-electrode system with Pt wire as counter and reference electrode. Both transmissive^{26,27} and reflective^{28,29} electrochromic devices were demonstrated previously for different target applications. For example, transmissive electrochromic devices can be used for antiglare mirrors and smart windows, and reflective devices are suitable for nonemissive display applications like e-paper, signage, and active camouflage. Reflective spectra of the stretchable device in bleached (-0.5 V) and colored (-1.8 V) states are shown in Figure 3a. The reflectance in the bleached state was normalized to 100% as the baseline and compared to that in the colored state. Reflections were suppressed within 400–900 nm but enhanced within 300–400 nm upon coloration, which is consistent with the deep violet/blue color observed. A reflection peak at 350 nm was observed. In previous studies, the observed transmission peaks for WO_3 at colored states was in the range of 400–500 nm and

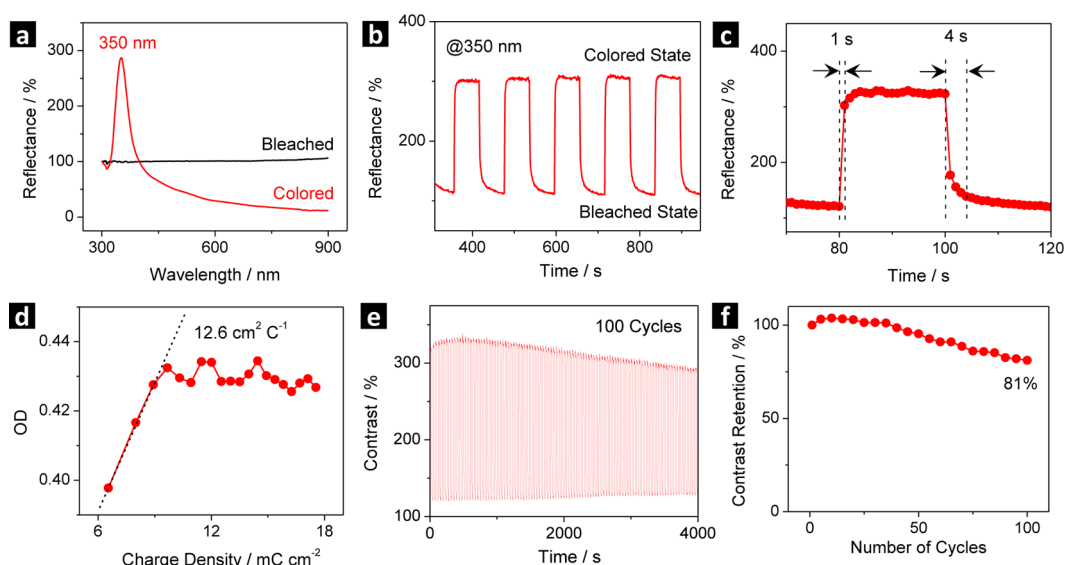


Figure 3. Electrochromic performances of the stretchable devices at relaxed state. (a) Reflective spectra of the stretchable electrode in colored and bleached states. (b) Switching behaviors measured at 350 nm. (c) Enlarged view of a single switching cycle showing the fast coloration and bleaching time. (d) Plot of OD versus charge density for CE calculation. (e) Cyclic switching for 100 cycles. (f) Stable cycling behaviors with 81% contrast retention after 100 cycles.

varied with applied potentials.^{12,26,27} The reflection peak for our stretchable electrodes shows a slight blue shift as compared with those observed in transmission mode, probably due to the reflection contributions from underlying AgNWs. Representative *in situ* switching behaviors of the stretchable device measured at 350 nm are shown in Figure 3b. Although the performances varied slightly within a batch of devices, fast and stable color switching was successfully achieved. For example, an enlarged view of a single switching cycle is shown in Figure 3c, and the measured coloration and bleaching time (defined as the time required to reach 90% of the full response) are 1 and 4 s, respectively.

Coloration efficiency is one of the most important characteristics of electrochromic devices. Higher CE is desired to use less charge or energy to achieve larger optical modulations. Moreover, cation insertion/extraction leads to crystal structure deformations and affects the long-term electrochemical cyclic stability, thus a reduced content of ion insertion/extraction is of interest to improve the device stability for practical applications. The efficiency is defined as the change in optical density (OD) per unit charge density (Q/A) and can be calculated based on $CE = \Delta OD / (Q/A)$, where $OD = \log(R_{\text{color}}/R_{\text{bleach}})$, Q is charge amount, A is electrode area, R_{color} is the reflectance in the colored state, and R_{bleach} is the reflectance in the bleached state. Figure 3d shows the relationship between *in situ* OD and intercalation charge density during a typical coloration process at -1.8 V. The CE value fitted from the linear region of OD–charge density curve is $12.6 \text{ cm}^2 \text{ C}^{-1}$. Previously, the switching behaviors for transmissive WO_3 electrochromic devices were usually monitored in the wavelength range of 550–750 nm (in most cases ~ 633 nm), where the highest contrast was obtained.^{12,24,26,27} Herein the highest contrast was obtained at 350 nm, as evident from the reflective spectra shown in Figure 3a. The preliminary result of $12.6 \text{ cm}^2 \text{ C}^{-1}$ for our reflective devices is relatively low as compared with those for transmissive devices ($16\text{--}100 \text{ cm}^2 \text{ C}^{-1}$)^{27,30} and can be further improved by optimizing the WO_3 fabrication processes since CE

was shown to be highly dependent on WO_3 morphology, structures, and crystallinity.^{26,27,31}

Electrochemical cyclic stabilities of the stretchable devices are shown in Figure 3e,f. The device maintained their functionalities after 100 cycles (Figure 3e) with 19% contrast degradation (Figure 3f). Possible reasons for the contrast degradation might include the following: (1) Amorphous WO_3 is slightly unstable in acidic electrolytes.¹² Part of the contrast degradation may arise from the dissolution of active materials. (2) Although the polarization potentials (colored at -1.8 V, bleached at -0.5 V) were selected to avoid the oxidation of AgNWs (Figure S3, Supporting Information), slight oxidations might still occur, especially after long-term cyclic testing. The oxidized silver with dark color may lead to reduced contrast.

Electrochromic performance variations upon stretching were also studied. Reflective spectra and switching behaviors of the stretchable devices in relaxed and stretched states are compared in Figure 4a,b, respectively. Optical images and detailed schematic diagrams of the stretching setup can be found in Figure S5, Supporting Information. The stretchable device was mounted onto a glass slide and could be stretched and fixed using two binder clips to fit in the UV–vis spectrophotometer chamber. No binder clips were used for property testing in Figure 3 at relaxed states. The reflective spectra collected using stretching setup (Figure 4a) are qualitatively similar to those in Figure 3a. Enhanced reflections in the ultraviolet region (300–400 nm) were also observed, but the relative intensity was reduced. The binder clips in the stretching setup introduced additional air gaps (~ 5 mm) between the stretchable device and the detection window of the spectrophotometer. Thus the reflective light was more severely scattered before being detected especially in the short wavelength region (300–400 nm), leading to the observed contrast reduction. As compared to that at relaxed state, the contrast was slightly reduced upon stretching. For example, the contrast at 633 nm is 56% at relaxed state and 50% at stretched state (Figure 4a). Switching behaviors of the devices monitored at

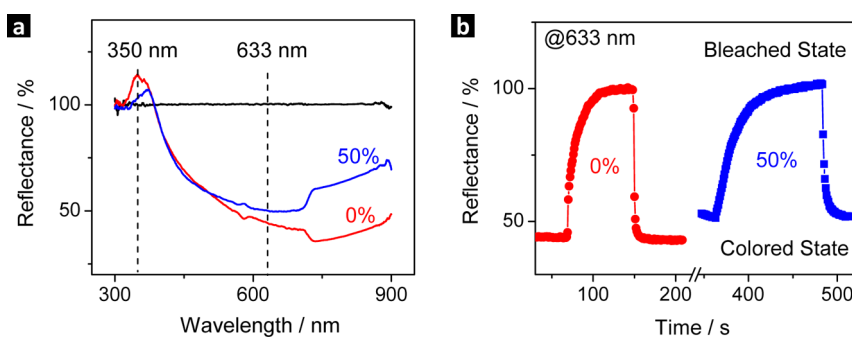


Figure 4. Compare the (a) reflective spectra and (b) switching behaviors of the electrochromic devices at relaxed (0%) and stretched (50%) states.

633 nm (most commonly used comparing wavelength for transmissive devices) in relaxed and stretched states are shown in Figure 4b. Note that the lower contrast in Figure 4b is at colored states, which is different from that in Figure 3b. It is evident that the stretchable devices maintained their functionalities at 50% strain, and the device can be repeatedly and reversibly colored/bleached at both relaxed and stretched states. The coloration/bleaching processes slowed down upon stretching. For example, the coloration time increased from 4 to 9 s when the device was stretched from 0 to 50% strain (Figure 4b). Analogous increase in bleaching time (from 25 s at 0% strain to 43 s at 50% strain) was also observed. The slower processes at stretched states can be understood by the electrode resistance variations. Electrode resistances would increase upon stretching and consequently result in smaller charge/discharge currents since the devices were polarized at fixed voltages. Thus, it would take a longer time to fully charge/discharge the active WO_3 film at stretched states.

Apart from electrochemical stability, mechanical stability against repeated stretching is another important characteristic of stretchable electrochromic devices. The unique embedded structures of the stretchable AgNW electrodes ensure outstanding robustness and durability against repeated stretching and even surface scratching and adhesive forces. The electrode resistance increased upon stretching due to the inter-sliding of percolative NW networks and possible loss of contact nodes.²⁰ Typically, the electrode resistances at respective strains became stable after 30 stretching cycles (Figure S5, Supporting Information). Unlike those stretchable electrodes with conducting materials on top of the elastic substrates,^{18,32} embedded structure provides outstanding durability even against Scotch tape adhesive forces as shown in our previous work.²⁰ Mechanical stability of the electrochromic devices were also tested as shown in Figure S6, Supporting Information. The reflective spectra and switching behaviors in Figure S6c,d were measured after stretching the device to maximum strain of 50% for 30 cycles. Reduced contrast at especially 50% strain was observed as compared with those before stretching cycling. Partial delamination of the active WO_3 layer into the electrolyte was observed, which resulted in the weakened switching behaviors. Further work on improving the adhesion stability is in progress. The adhesion of active electrochromic layer to the underlying substrate can be effectively improved by surface functionalization as shown in our latest work,³³ which may also be applicable to stretchable devices. The adhesion stability can also be improved by optimizing the WO_3 layer thickness. Thicker WO_3 layers are more rigid and tend to delaminate upon mechanical deformation, although they provide better contrasts. Proper balancing and the detailed relationships between

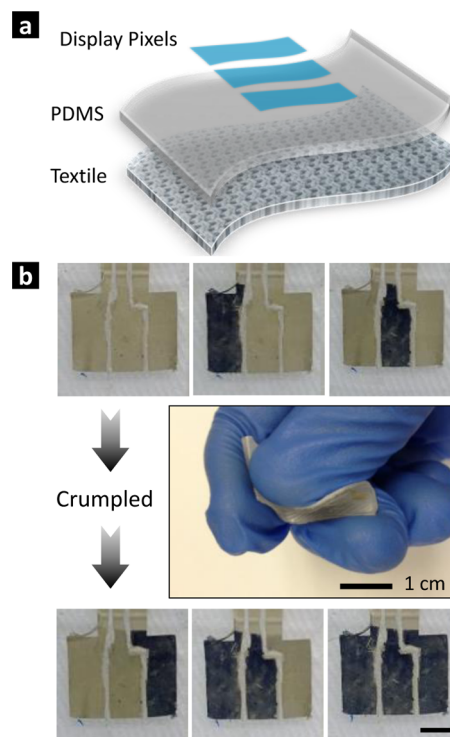


Figure 5. (a) Schematic of the electrochromic electrode implanted onto wearable textiles. (b) Example images showing the capability to control the coloration/bleaching of individual display pixels and their mechanical stabilities against deformations such as crumpling. Scale bar for display pixels: 2 mm.

active layer thickness and optical contrasts are of interest for further studies.

One of the most exciting applications of stretchable electrochromic devices is for future wearable and implantable displays. The soft display panels with individually addressable pixels could potentially be implanted on clothes or skin for the display of stationary and even moving texts and images. As a proof of concept, wearable electrochromic devices implanted on a cotton textile substrate are shown in Figure 5. Textile substrates, analogous to papers, possess 3D woven structures with rough surfaces^{34–36} which make the direct implantation of patterned AgNW display pixels difficult. In paper electronics, a polymer interlayer such as parylene is usually employed to passivate the paper substrates and improve their physical and chemical properties for the following lithography processes.³⁷ Herein we employ a layer of PDMS to facilitate the implantation of patterned AgNW display pixels, as shown in Figure 5a. Representative example images of the wearable electrochromic device are shown in Figure 5b. The three individually addressable display pixels can be colored and bleached individually or in any combinations. The wearable electrochromic devices are mechanically robust and could maintain their functionalities after various forms of deformations such as crumpling

(Figure 5b). Although only three pixels were demonstrated in our prototype, scale fabrication of display pixel arrays can be readily achieved, paving the way for unprecedented applications such as wearable display panels implanted on clothes and skin.

CONCLUSIONS

In conclusion, stretchable and wearable WO_3 electrochromic devices were fabricated based on AgNW elastic conductors. The stretchable devices are mechanically robust and can be stretched, twisted, folded, and crumpled. Deep violet color was observed for the stretchable electrodes in colored states, which is consistent with the reflection spectrum showing enhanced reflectance within 300–400 nm. Fast coloration (1 s) and bleaching (4 s) time and good cyclic stability (19% contrast degradation after 100 cycles) were successfully achieved at relaxed states. The stretchable device can maintain their functionalities

even when stretched up to 50%, although the switching processes slowed down due to the resistance variations. Wearable electrochromic electrodes implanted on cotton textile was demonstrated. The display pixels can be controllably colored and bleached individually or in any combinations. The electrochemical and mechanical stabilities can be further improved in future studies. For example, since AgNWs are chemically reactive, the search for alternative conducting materials with proper chemical, electrical, and optical properties is of interest to improve the electrochemical stability. Mechanical stability against repeated stretching can potentially be improved by surface functionalization and active layer thickness optimization. Unlike existing technologies based on rigid substrates, the stretchable and wearable electrochromic devices may find broad applications in next-generation soft displays and related devices.

METHODS

Stretchable Electrode Fabrication. The stretchable electrodes were fabricated *via* a lithographic filtration method, as described in our previous work.²⁰ PDMS base and curer (Dow Corning, Sylgard 184) was mixed (weight ratio 10:1), degassed, and cured at 60 °C for 2 h. Soft filtration masks with desired patterns were prepared from the cured PDMS membrane (~1 mm). The mask was put on top of a polycarbonate (PC) filter membrane (Millipore GTTP, pore size 220 nm) for filtration. Commercially available AgNWs (Seashell Technology LLC, USA) with diameters of 40–100 nm and lengths of 20–100 μm were dispersed in ethanol (1 mg mL^{-1}). Uniform and well-defined AgNW patterns were obtained after filtration. Liquid PDMS was poured on top of the filter membrane with NW patterns and then cured using the same procedure for mask fabrication. The solidified PDMS membrane was peeled off from the PC membrane after curing, and the NW patterns were successfully transferred to the PDMS elastomer matrix.

Wearable Electrode Fabrication. Wearable electrodes with patterned AgNW display pixels were fabricated on a cotton fabric substrate. A piece of fabric (100% cotton, CKC Uniform, Singapore) was covered on top of the degassed liquid PDMS before curing. The thin layer of PDMS was used to combine the AgNW pixels and cotton substrate. The AgNW patterns were then successfully transferred to the fabric substrate after PDMS curing.

WO_3 Deposition. Electroactive WO_3 layer was deposited onto the stretchable and wearable electrodes using a three-electrode system, with Pt plate and Ag/AgCl as counter and reference electrodes, respectively. The source solution was prepared as described previously.³⁸ Tungsten power (1.8 g, Alfa Aesar, USA) is mixed with H_2O_2 solution (30%, 60 mL, Tokyo Chemical Industry, Japan) and stirred constantly for 12 h. The solution became yellowish after stirring, and the sediments were filtered to obtain a clear solution for WO_3 deposition. The electrochemical deposition was performed at -0.5 V for 200 s using an electrochemical analyzer system (Autolab Potentiostat, PGSTAT302N).

Electrode Characterizations. Microstructures of the stretchable electrodes were characterized using SEM (JSM 7600F). A home-built stretching stage was used to measure the resistance variations upon stretching (Figure S4, Supporting Information). Electrochromic properties were measured using a two-electrode system with Pt wire as counter and reference electrode in 0.5 M H_2SO_4 electrolyte. The stretchable electrode was mounted

onto a glass slide, and a PDMS casing was used to contain the electrolyte for testing. Two binder clips were used to fix the electrode at specific strains (Figure S5, Supporting Information). The measurements were carried out using Solartron (model 1470E) with polarized square-wave potentials of -1.8 and -0.5 V *versus* reference electrode. Reflectance spectra were measured using UV-vis spectrophotometer (Shimadzu UV-3600) equipped with a reflective light sensor.

Conflict of Interest: The authors declare no competing financial interest.

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Supporting Information Available: Additional materials mentioned in main text. This material is available free of charge *via* the Internet at <http://pubs.acs.org>.

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