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RESEARCH ARTICLE

Fabrication of agarose hydrogel with patterned silver nanowires for motion sensor

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Abstract

In this work, a facile strategy is proposed to construct stretchable electronics based on agarose hydrogels. The hot agarose solution is casted onto a template with patterned Ag nanowires, endowing agarose hydrogel with patterned conductive surface. After further heating treatment, Ag nanowires can be embedded into the agarose hydrogel, which improves the stability of Ag pattern and has no obvious effect on the conductivity of hydrogels. The agarose hydrogel with patterned Ag nanowires is certified to be an effective stretchable electrode to record the motion of joints, which has great potential applications in the field of wearable devices.

Keywords Agarose hydrogel · Ag nanowire · Stretchable electronics · Pattern · Casting strategy

Introduction

Recently, development of wearable electronics has given rise to great opportunities in a wide range of applications for personal health care, which could collect and measure biophysical (skin temperature, bioelectricity, etc.) and biochemical (electrolytes, metabolites, etc.) signals created by physiological processes conveniently and quickly [1]. Traditional electronic systems, which are constrained to rigid and flat formats, are too rigid to conform to human skin surface or bear intense bending and stretching [2–4]. Up to date, bendable, compressible, stretchable, conformable and deformable electronics to arbitrary body shapes is more able to meet the need of human [5–8]. Until now, many compounds, such as polydimethylsiloxane, polyvinyl alcohol, polyurethane and polyacrylic acid, have been used as substrates to construct wearable devices successfully with an excellent compressible and stretchable ability. However, those materials more or less suffer from cytotoxicity to human body due to the cross-linkers and initiators. To solve this issue, hydrogels with excellent biocompatibility and similar properties of body tissue formed by natural compounds (such as gelatin, collagen, alginate, chitosan and agarose) have been developed to fabricate wearable devices [9–11]. In particular, agarose, a natural polysaccharide derived from algae, is an ideal candidate as a substrate to construct wearable devices. Agarose can form stable and robust hydrogels with biochemical and mechanical properties at very low concentration (0.1%, wt%) below 40 °C [12]. Compared with other hydrogels, agarose gels have the merits of highly porous structure, high hydrophilicity, free of charged groups and rich hydroxyl groups on polysaccharide chains, which have been used widely in the fields of tissue engineering and repairing, drug release and protein

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separation [13–21]. Unfortunately, agarose hydrogels are electrically neutral materials, requiring to be integrated with conductive materials to improve its conductivity. Graphene, carbon nanotubes and metal nanowires are the popular conductive materials, which have been selected to dope within hydrogels and promote the conductivity to satisfy requirements of wearable devices. Although carbon materials are hot research topics in the field of wearable devices recently, the poor electrical conductivity of carbon materials battles their further development and application [22, 23]. To develop a facile and low-cost strategy, constructing wearable devices with excellent biocompatibility and conductivity is still desirable.

Silver nanowires (Ag NWs) are selected as conductive materials to elevate conductivity of hydrogels because of their higher conductivity than carbon nanomaterials or any other metal nanowires, which is at least one order and three orders of magnitude higher than single-walled nanotubes (SWNTs) and graphene, respectively [24–28]. In addition, Ag NWs were certified to have good mechanical flexibility suitable to improve conductivity and strength of hydrogels [29–31]. In this work, a "pattern and casting" strategy has been proposed to endow agarose gels with great conductivity. The hot agarose solution was casted onto a template with patterned Ag nanowires, endowing agarose hydrogels with patterned conductive surface. After further heating treatment, Ag NWs could be embedded into agarose hydrogel, which improved stability of nanowires pattern and kept conductivity of hydrogels (Fig. 1). The hydrogels with patterned Ag NWs were demonstrated to be an effective wearable sensor for human motion detection.

Materials and methods

Materials

Agarose (Agar, type XI) and polyvinylpyrrolidone (PVP, Mr = 360 000) were purchased from Sigma-Aldrich (USA). Silver nitrate ($AgNO_3$), ethylene glycol (EG) and ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) were purchased from Sinopharm Group Chemical Reagent Co., Ltd. All chemicals were reagent grade.

Methods

Synthesis of silver nanowires (Ag NWs)

Ag NWs were prepared by a one-pot method. 0.4 g PVP was added to 50 mL ethylene glycol (EG) and dissolved by magnetic stirring at room temperature. Afterward, 0.5 g of $AgNO_3$ was added to PVP solution with magnetic stirring in dark, until a transparent solution was obtained. Finally, 7.0 g of a $FeCl_3$ salt solution (600 μM in EG) was added to the mixture quickly and stirred for 90 s. Then, the mixture was immediately transferred into a reactor preheated at 130 °C to grow Ag NWs for 5 h. Finally, acetone was used to wash precipitates with centrifugation of 4000 rpm for 5 min, and then, ethanol was used to wash precipitates with centrifugation of 8000 rpm for 5 min. Finally, Ag NWs were re-dispersed in ethanol for future use.

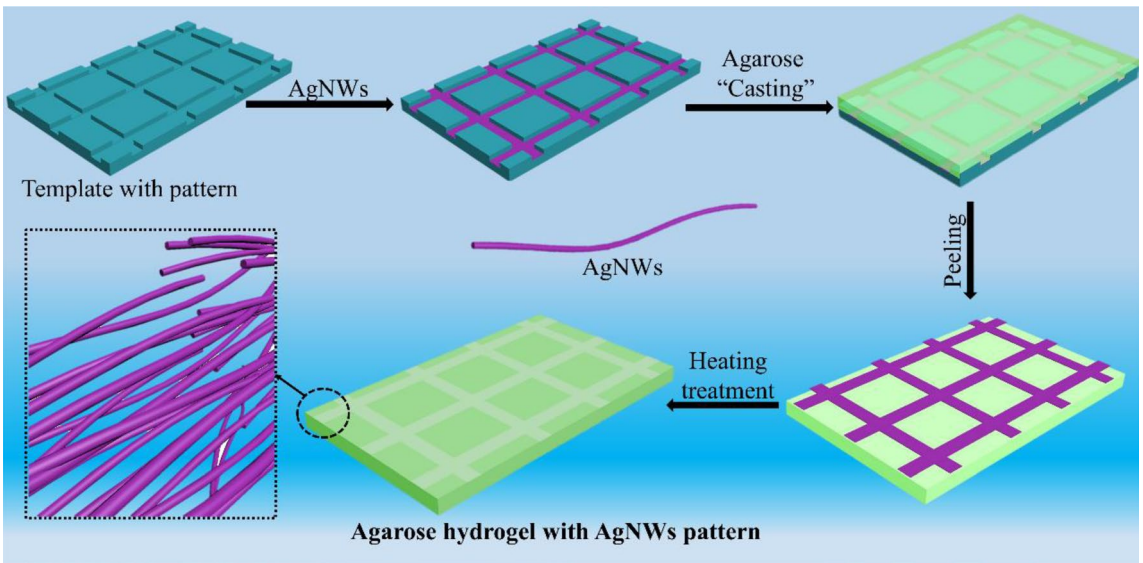

Fig. 1 Schematic illustration of how to prepare agarose hydrogels with patterned Ag NWs

Preparation of templates with patterned Ag NWs

The patterned Ag NWs were realized by a PVC mask template (Meishang Plastic, Guangzhou, China). The thickness of mask template was 80 μm. The mask was first pasted onto a pre-cleaned substrate. The substrate could be silicon wafer, glass or plastic materials. Then, the Ag NWs suspension was drop-casted onto the mask template. The patterned Ag NWs were obtained after ethanol volatilizing completely.

Casting of agarose solution into templates

Sixty milligrams agarose was incubated in a deionized water bath at 95 °C for 15 min to obtain transparent solution. Then, the hot agarose solution was immediately casted onto the mask template. The hydrogel film could form after cooling to room temperature. The free-standing agarose hydrogel film with patterned Ag NWs could be easily peeled from templates.

Heating treatment of hydrogels

The agarose hydrogel film with patterned Ag NWs was placed 10 cm away from the infrared lamp (250 W) and treated for 0, 5, 10 and 15 min.

Motion signal recorded by hydrogels sensor

The real-time electrical signals of the strain sensor based on the resistance changes of hydrogels in a different state were recorded by CHI 660E equipment. The relative change in resistance was calculated by Ohm's law ($R = U/I$) on the basis of the application of a constant voltage to the strain sensor-induced changes in electrical currents under different strains.

Results and discussion

Synthesis of Ag NWs

Silver nanowires with an average width of 60–80 nm and length of 50–100 μm were fabricated by a simple and

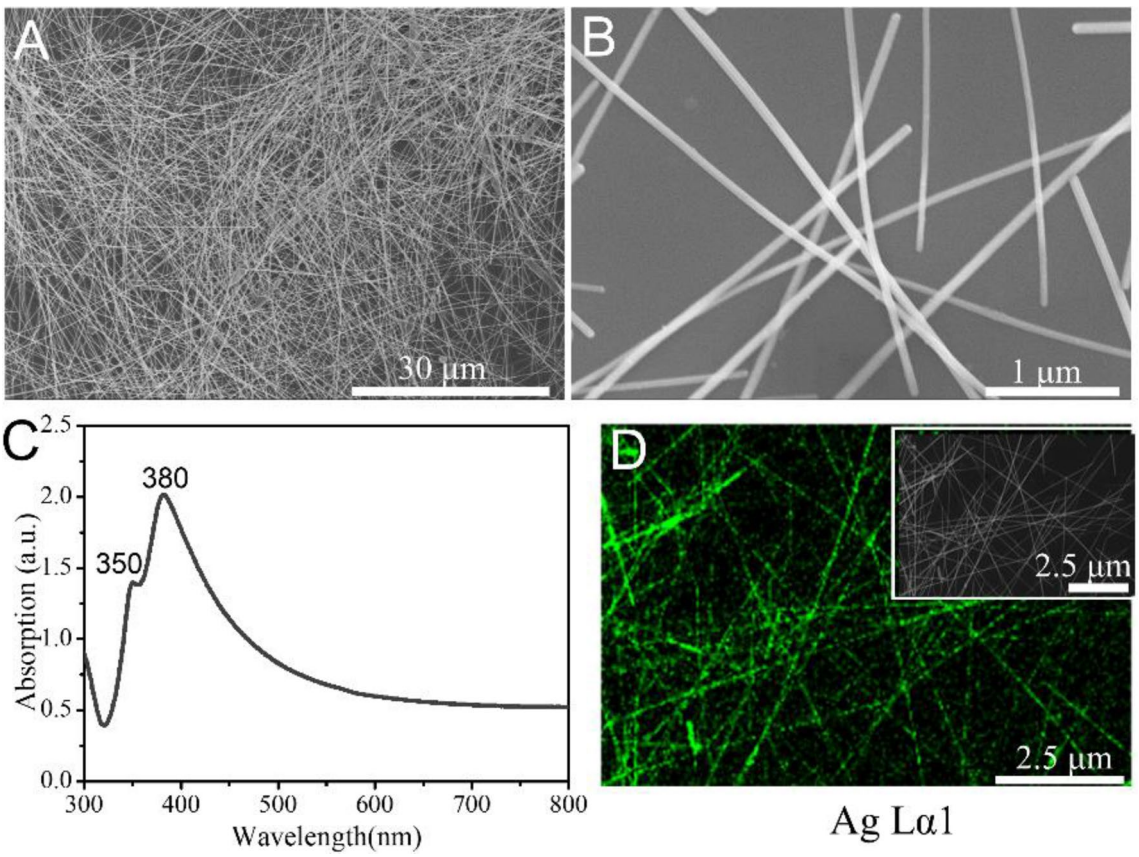

Fig. 2 **a** SEM image of formed Ag NWs; **b** the corresponding SEM images with high magnification; **c** UV–Vis of Ag NWs in ethanol solution; **d** element mapping of Ag NWs

straightforward polyol process method according to previous works [32, 33]. As shown in Fig. 2a, b, prepared Ag NWs had an excellent distribution and uniform size, which were robust enough to withstand separation process from reaction media. No obvious changes in morphology and length were observed during the process. Meanwhile, UV–Vis spectra displayed that Ag NWs had a characteristic absorption peak located at 350 and 380 nm (Fig. 2c), ascribing to the bulk and transversal plasmon, respectively [34–36]. In addition, the element mapping of Ag NWs demonstrated the nanowires were comprised of silver (Fig. 2d) only, indicating the purity of Ag NWs. It should be noted that Ag NWs were fabricated in hot polyol solution, which promised the hydrophilicity of nanowires. The formed Ag NWs could distribute in ethanol solution well, benefiting attachment to hydrophilic agarose hydrogels.

Casting agarose solution into template with patterned Ag NWs

Agarose was a linear polysaccharide consisted of alternating *D*-galactose and 3,6-anhydro-*L*-galactopyranose units linked by α-(1 → 3) and β-(1 → 4) glycosidic bonds [37]. Agarose hydrogels have been investigated for cell culture and tissue engineering because of the excellent biocompatibility. At a high temperature, agarose formed random coils in aqueous solution. After cooling below 40 °C, agarose molecules formed both single and double helices, bundling and assembling into fibrillar networks to form hydrogels [38]. Herein, a "pattern and casting" strategy was used to prepare conductive agarose hydrogels. As shown in Fig. 1, Ag NWs distributed in ethanol with different concentrations were dropped into the pattern on template. Upon evaporation of Ag NWs medium, the hot agarose solution was casted onto the same pattern.

After cooling down to 25 °C, agarose hydrogels with patterned Ag NWs could be peeled from templates, which could be taken as conductive materials for the following construction of stretchable electronics. It should be noted that the whole process was performed in aqueous or ethanol medium, avoiding the possible contamination of toxic organic solution, which promised a comfortability to human skin. The agarose hydrogels with patterned Ag NWs were assayed by optical microscope due to their good transparency. As depicted in Fig. 3a, Ag NWs could be observed clearly and distributed well on the surface of hydrogels without aggregation, which was related to high hydrophilicity of Ag NWs [39]. SEM image in Fig. 3b gives a similar result,

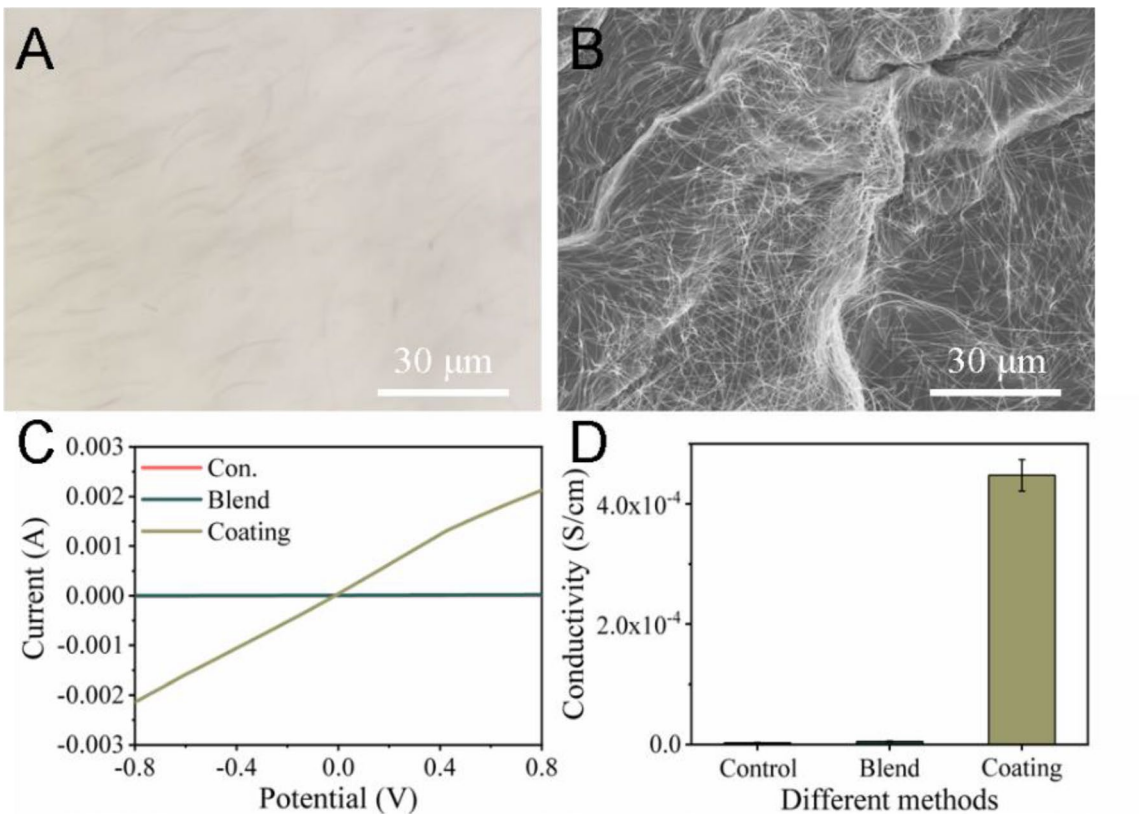

Fig. 3 **a** Optical image of hydrogels with patterned Ag NWs; **b** the corresponding SEM images; **c** and **d** the conductivity of hydrogels with patterned Ag NWs

showing Ag NWs attached to the surface of hydrogels and formed a network structure whose junctions led to conductivity of the hydrogels. The thickness of patterned Ag NWs was ~ 5 μm (Figure S2). Meanwhile, the morphology and length of Ag NWs had no obvious change during the "casting" and peeling process, indicating a high stability of Ag NWs.

Good conductivity of hydrogels was critical to construct effective wearable devices. The measurement of conductivity of hydrogels with patterned Ag NWs was taken, as shown in Fig. 3c, d. The obtained hydrogels possessed a fine conductivity (Voltage 0.8 V, conductivity 4.1×10^{-4} S/cm, Fig. 3d) compared with a control group (without Ag NWs) which was almost non-conductive. In addition, hydrogels with the same content of Ag NWs by homogeneous blending method were taken as another control, which displayed a poor conductivity as shown in Fig. 3c, d. It is indicated that the "pattern and casting" strategy could endow hydrogels with excellent conductivity, minimizing the used amount of Ag NWs. Meanwhile, the conductivity of hydrogels with pattern Ag NWs could be adjusted by the concentrations of nanowires (Figure S1), benefiting the regulation of physicochemical properties to match requirements of different wearable devices.

Heating treatment of hydrogels with patterned Ag NWs

Agarose hydrogels were thermosensitive, which could change from gel to sol state after heating treatment. To further improve the stability of Ag NWs pattern on the surface of hydrogels, heating treatment by infrared lamp (250 W) was carried out to melt hydrogel surfaces, which would induce embedding of Ag NWs into hydrogels. As shown in Fig. 4, network of Ag NWs became tightly connected and was buried in the hydrogels along with increasing time of heating treatment, giving rise to a strong physical adhesion to hydrogels. In addition, no obvious change in morphology and length of Ag NWs appeared during the whole treatment. The time of heating treatment was fixed at 10 min, because the surface of agarose hydrogels had cracks and wrinkles caused by evaporation of water from hydrogels beyond 10 min. The high magnified SEM images in Fig. 5a displayed clearly that the nanowires at the top of the surface were fully or partially embedded into hydrogels after heating, which promoted the stability of patterned Ag NWs on the surface.

The wear-resistant experiments were carried out on the UMT-3 (Burker, USA) at a test rate of 120 mm/min. Figure 5b demonstrates a great improvement in stability of patterned Ag NWs on the surface of hydrogels after heating

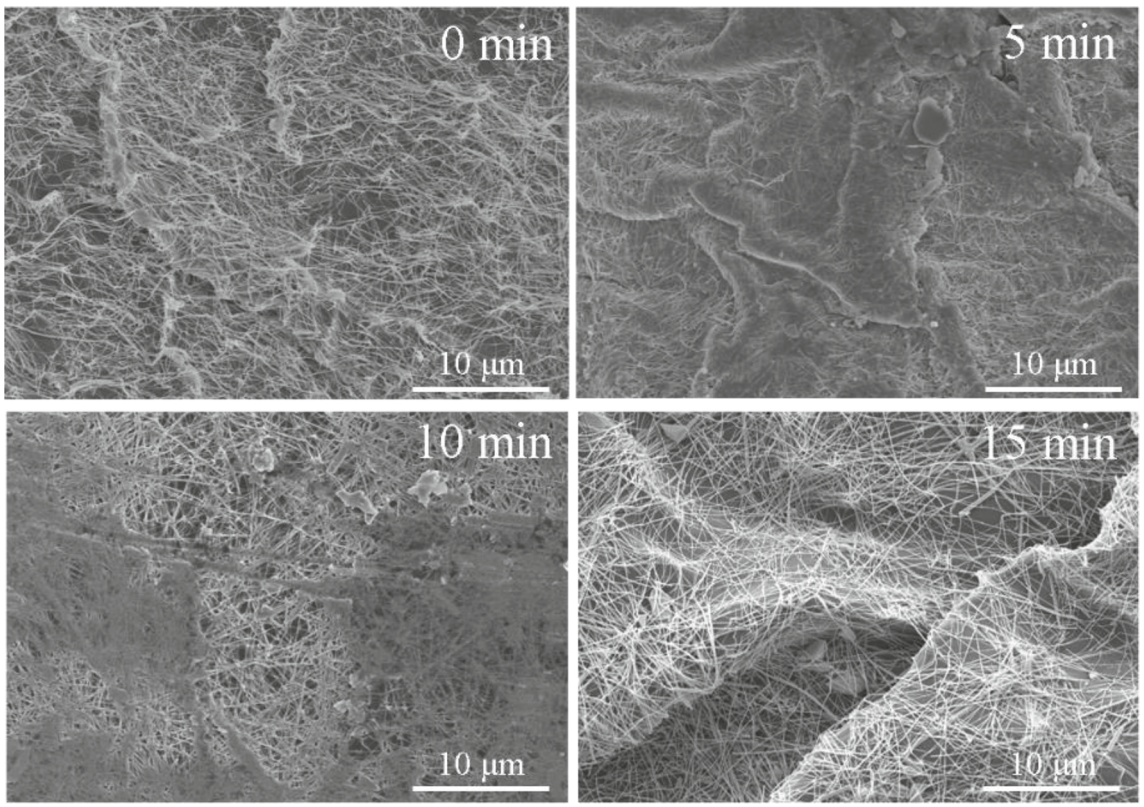

Fig. 4 SEM images of heating treatment of agarose hydrogels with different time (0, 5, 10, 15 min)

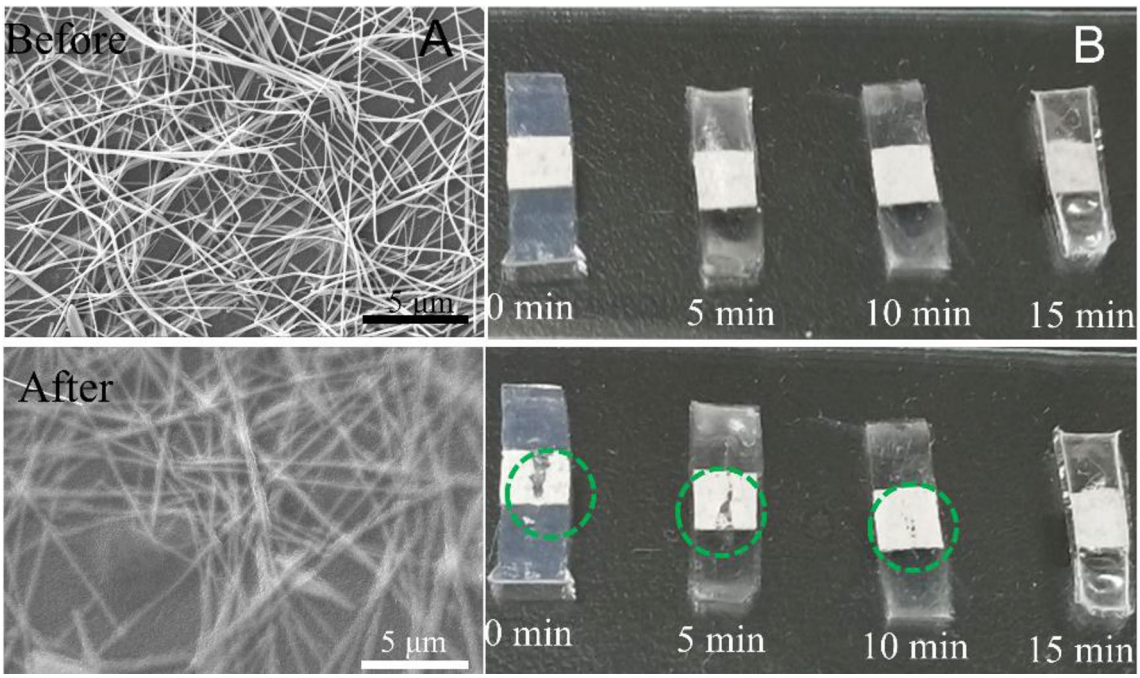

Fig. 5 **a** The high magnified SEM images of heating treatment of agarose hydrogels with 15 min; **b** the wear-resistant experiments of agarose hydrogels with patterned Ag NWs heated by different time (0, 5, 10 and 15 min)

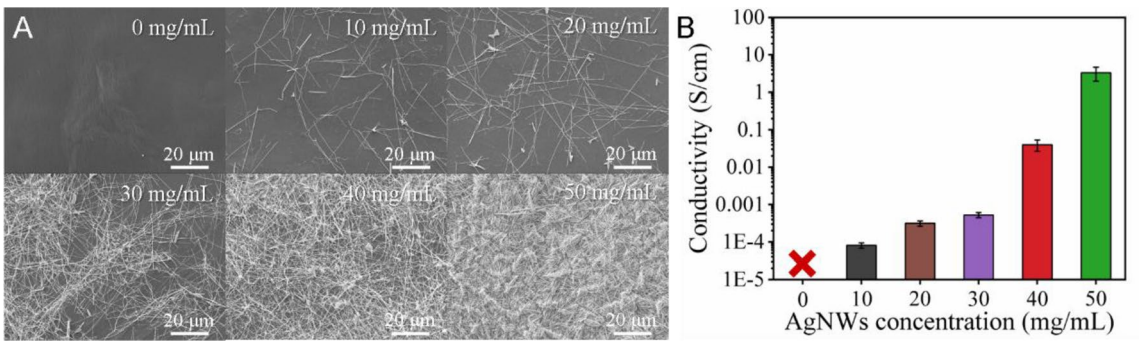

Fig. 6 **a** SEM images of heating treatment of agarose hydrogels containing different amounts of Ag NWs (0, 10, 20, 30, 40 and 50 mg/mL); **b** the corresponding conductivity of the samples

treatment. The samples (with 10 min of heating treatment) possessed enough wear resistant.

To test changes in conductivity of hydrogels with Ag NWs after heating treatment, the conductivity of hydrogels with different contents of Ag NWs was investigated in a detail. As displayed in Fig. 6a, b, all the samples showed excellent conductivity which was elevated along with increasing Ag NWs content. It is demonstrated that the conductivity of hydrogels could be turned by contents of Ag NWs. Furthermore, there was no obvious change in conductivity of hydrogels compared with the one without heating treatment (Figure S1).

Motion signals recorded by flexible electrode of hydrogel

To demonstrate potentials of the "pattern and casting" technique and conductive films for stretchable electronics, a simple circuit with an LED light was designated and constructed on the top of agarose hydrogels as shown in Fig. 7a, b. It was observed that LED light was on after exerting voltage of 3 V (Fig. 7b), indicating good conductivity of hydrogels with patterned Ag NWs.

To further test a possibility of agarose hydrogel as a motion sensor, the relationship between resistance and bending of finger joints was recorded and investigated in a detail.

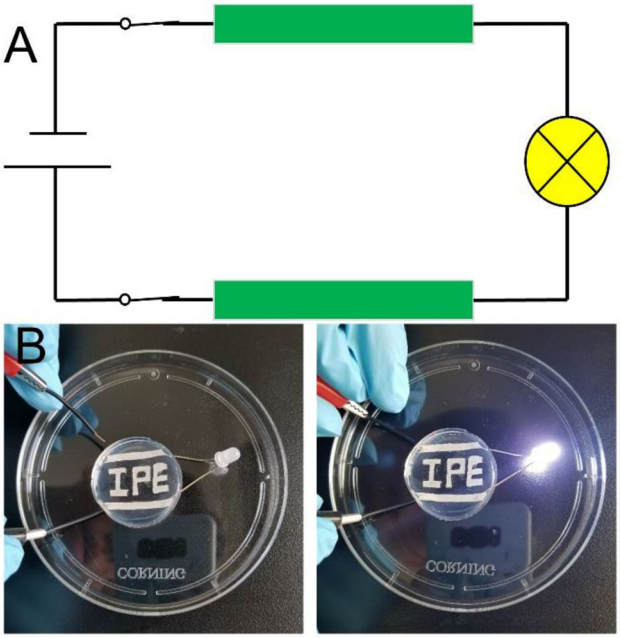

Fig. 7 **a** Schematic illustration of the designed circuit with an LED light; **b** photographs of the circuit constructed on surface of agarose hydrogels

As depicted in Fig. 8a, regular relative change in resistance (R/R_0, R, resistance of the electrode at different bending angles of finger; R_0, resistance of the electrode at bending angle of 0°) was found, indicating a reversible and rather stable change in the resistance during whole experiments. Furthermore, agarose electrodes were attached onto lateral and medial wrist and elbow joint to detect human motions. The results in Fig. 8b–d clearly illustrated the resistance changes in electrodes with good reversibility and stability during three bending–releasing cycles. Therefore, the sensor could be employed for accurate motion of human, indicating the sensor provides the potential applications in the field of flexible wearable devices.

Conclusions

Herein, a stretchable electronic device with excellent biocompatibility and effective performance is designed and constructed on the surface of agarose hydrogels through a facile strategy of "pattern and casting." During the fabrication process, the hot agarose solution is casted onto the template with patterned Ag NWs, endowing agarose hydrogels with patterned conductive surface. After peeling from the

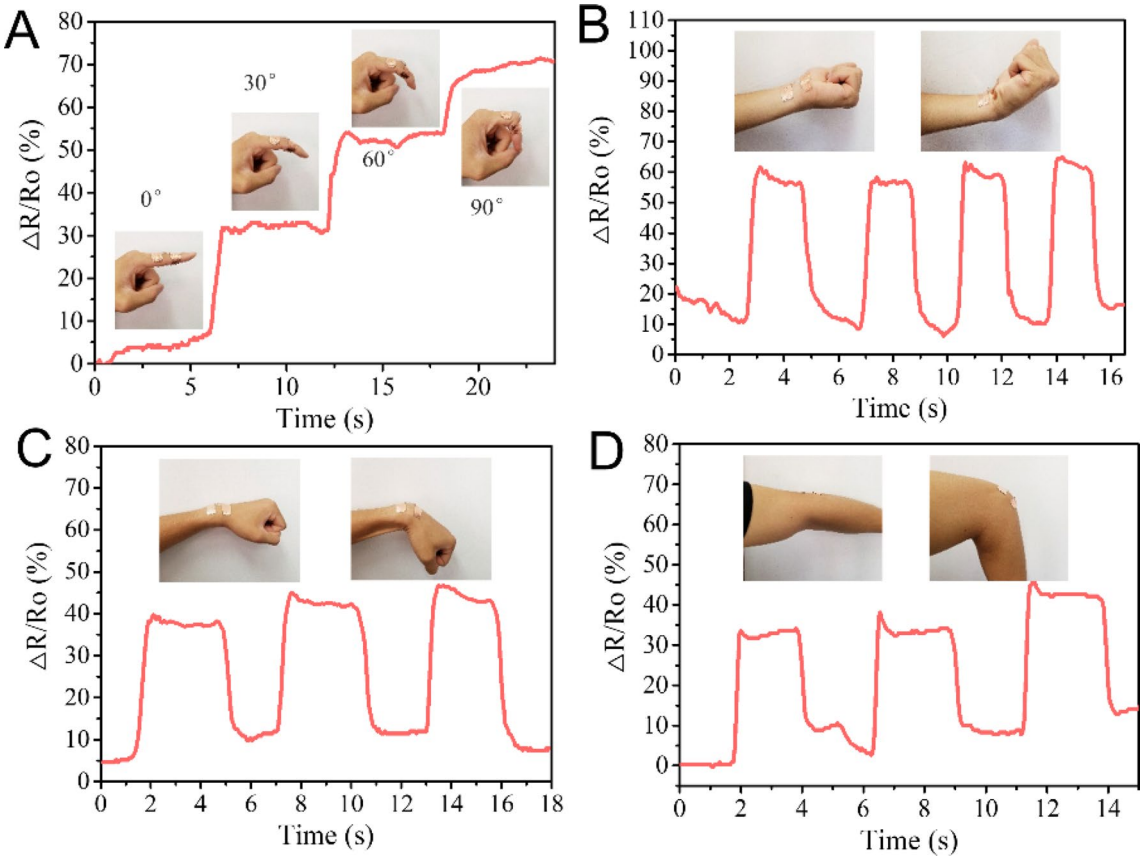

Fig. 8 **a** Detection of finger joint motion by agarose hydrogels electrode; **b** bending motion of medial wrist; **c** lateral wrist; **d** elbow joint

template and further irradiation of infrared lamp, Ag nanowires can be embedded into hydrogels due to the melting of agarose, which improves the stability of nanowires pattern and has no obvious effects on the conductivity of hydrogels. The agarose hydrogels with patterned Ag nanowires are certified to be an effective and stretchable electronics for motion detection and have great potential applications in the field of wearable devices.

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Compliance with ethical standards

Conflict of interest The authors declare that there is no conflict of interest.

Ethical approval This study does not contain any studies with human or animal subjects performed by any of the authors.

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