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Soft piezoresistive pressure sensing matrix from copper nanowires composite aerogel

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Abstract We report on a simple yet efficient approach to fabricate soft piezoresistive pressure sensors using copper nanowires-based aerogels. The sensors exhibit excellent sensitivity and durability and can be easily scalable to form large-area sensing matrix for pressure mapping. This opens a low-cost strategy to wearable biomedical sensors.

Keywords Copper nanowires · Aerogel · Piezoresistive · Pressure sensing matrix

1 Introduction

Soft flexible pressure sensing devices can have a plethora of technical applications in future, ranging from electronic skins [1–3], energy harvesting from motions [4, 5], flexible touch display [6], implantable and wearable electronics [7–10]. The key requirement is to integrate compliant mechanics with outstanding optoelectronic properties into one single multifunctional system, which is often challenging to realize with traditional rigid inorganic bulk materials. Recently, various nanomaterials, including metallic and nonmetallic nanowires [8, 11–13], ionic liquids [14, 15], carbon nanotubes [16–18], graphene [7, 19–21], polymer based materials [22, 23] had been reported as promising candidates in terms of fabrication of flexible pressure sensors. Despite the abundance of material choices, some of the material mentioned above involves tedious fabrication processes or scarcity of the material which results in higher production cost and depletion of materials [24–26]. In this context, copper nanomaterial is recently gaining popularity due to its low price and more abundance compared to indium tin oxide, yet possesses comparable conductivity to silver [25, 26].

Recently, we have demonstrated ultralight aerogel monoliths from copper nanowires (CuNWs) [27] and CuNWs-poly(vinyl alcohol) (PVA) composite aerogel monolith [11] using freeze drying techniques. The resulting aerogels could be used as waste oil clean up and stretchable conductors [11, 27]. Nevertheless, it has to be noted that the hydrazine-reduced CuNWs used in these demonstration have small aspect ratios [25, 27], hence limiting the elasticity of resulting aerogels. Highly stretchable sensors were not possible unless embedding them into polydimethylsiloxane (PDMS) [11]. Recently, a CuNWs synthesis approach had been reported producing CuNWs with aspect ratio higher than 4,000 without involving the use of highly hazardous hydrazine [28, 29]. These CuNWs are mechanically robust and highly electrical conductive, which emerge as a candidate in fabrication of transparent conducting film [29]. By combining these materials with rubbery polymer composites, this design would offer superior elasticity and mechanical robustness while remain electrically conductive.

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The composite aerogel has to be sealed to retard the oxidation of CuNWs and mechanically assisted by Ecoflex support to improve the life cycle due to cyclic loading. The presence of this support also helps to improve the sensitivity at higher pressure range. More importantly, this pressure sensor design could be scaled up to any number of pixels required as most of the fabrication processes are wet chemistry based and does not incur complicated processes. We believe that this pressure sensor has the capability of being used for medical purposes such as examine force exerted by a person's foot by orthopedist in order to diagnose foot pathologies such as diabetic foot and Plantar Fasciitis.

2 Materials and methods

2.1 Materials

Copper(II) chloride dihydrate ($CuCl_2{\cdot}2H_2O$, reagent grade), hexadecylamine (HDA, 90 % technical grade), and polyvinyl alcohol (PVA, $M_w = 85{,}000$–$124{,}000$) were purchased from Sigma-Aldrich. D(+)-Glucose (anhydrous for biochemistry Reag. Ph Eur) was obtained from Merck. DuPont™ Solamet® PV412 silver based polymer conductive paste was purchased from DuPont Microcircuit Materials. Ecoflex® supersoft 0030 platinum cure silicone rubber set were obtained from Smooth-On, Inc. SYLGARD® 184 Silicon Elastomer Curing Agent and SYLGARD® 184 Silicon Elastomer Base were obtained from Dow Corning. All of these chemicals were used as received. Stainless thin conductive yarn/thick conductive thread was obtained from Adafruit Industries.

2.2 Synthesis of CuNWs

1.8 g HDA (capping agent), 210 mg $CuCl_2{\cdot}2H_2O$ (precursor) and 100 mL water were mixed in a 250 mL Schott bottle, capped, heated and stirred at 100 °C and 600 r/min for 30 min. After the mixture turned into light blue in colour, 1 g D(+)-glucose was added into the mixture. The stirring speed was reduced to 350 r/min and allowed to react for 6 h. The mixture was allowed to cool for 15 min before purification. For purification, mixture was centrifuged at 6,000 r/min for 15 min. Then, the precipitated CuNWs were re-dispersed in 5 mg/mL PVA solution and sonicated for 15 min.

2.3 Fabrication of CuNWs-PVA-Ecoflex

In the first step, Ecoflex template was fabricated using three-dimensional (3D) printed pole structure. Briefly, precured Ecoflex, the mixture of the "Ecoflex part A" and the "Ecoflex part B" with a ratio of 1:1, was poured into a petri dish. The 3D printed poles structure was then inverted so that the poles were being embed into the Ecoflex mixture in order to create holes when the mixture was cured. The mixture of CuNWs and PVA poured into Ecoflex template and left in a freezer at −80 °C for 2 h. Then, the frozen sample was freeze-dried at a sublimation temperature of −85 °C and a pressure of 0.01 mbar (1 mbar = 100 Pa).

2.4 Sensor fabrication

The Cr/Au electrode (thickness at 5 nm/40 nm) were deposited onto PDMS substrates (40 mm × 40 mm) using a designed shadow mask by electric beam evaporator. The spacing between adjacent electrodes was 2 mm with the width of electrodes at 3 mm. Six contact pads of size 1 mm × 3 mm were deposited at the ends of the electrodes to establish external contact. The electrodes were coated with silver conductive paste to ensure a conformal contact between the electrode and CuNWs-PVA aerogel. The electrode coated PDMS and the bottom of the CuNWs-PVA-Ecoflex were treated by thin oxygen plasma (Harrick Plasma Cleaner PDC-001) followed by an irreversible bonding. The top electrode coated PDMS was oriented 90°, oxygen plasma treated and sealed to the top of the CuNWs-PVA-Ecoflex.

2.5 Instruments

CuNW-PVA aqueous solutions were frozen by a SANYO ultra-low temperature freezer at −80 °C, and the freeze drying process was performed with FreeZone 2.5 L Benchtop Freeze Dry system. The dimensions of pressure sensors were measured by a Stamvick caliper with an accuracy of 0.01 mm, and the weight of the samples were checked through a Mettler Toledo balance (MS 105DU) with an accuracy of 0.01 mg. Scanning electron microscope (SEM) images of CuNW-PVA composite aerogels were taken with FEI Nova NanoSEM™ 430 field emission gun SEM operated at an acceleration voltage of 3 kV and a working distance of 4–5 mm. Transmission electron microscope (TEM) images of CuNWs were taken with FEI Tecnai™ T20 operated at an accelerating voltage of 200 kV. The Cr/Au electrodes were deposited onto 40 mm × 40 mm PDMS substrates using a designed shadow mask by an electric beam evaporator (Intlvac Nanochrome II, 10 kV). The designed shadow mask and the template which used to fabricate Ecoflex template for aerogel were printed using 3D printer (Objet Eden260V). The mechanical properties measurements were done using SmarAct stepping positioner (SLC-1730) controlled by custom LabView program and force data measured by a GSO series load cell with capacity of 25 g (GSO-25)