



# Controllable assembly of silver nanoparticles based on the coffee-ring effect for high-sensitivity flexible strain gauges



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## ABSTRACT

An effective and facile strategy was developed to assemble silver nanoparticle (AgNPs) into a series of close-packed silver nanoparticles wires (AgNWs), and successfully construct a high-sensitivity strain gauge on the flexible PET substrate. In this work, AgNPs were directly ink-jet printed onto the flexible PET substrate and assembled into paralleled twin lines induced by the coffee-ring effect. By regulating the coffee-ring effect, a series of well-organized AgNWs were controllably fabricated with a line width of 5–6  $\mu\text{m}$ . Due to the exponential dependence of relative resistance variation to the applied mechanical strains, the assembled AgNWs-based strain gauge could achieve highly sensitive strain detection, exhibiting a high gauge factor approximately  $G = 50\text{--}60$  and a lowest strain level of 0.061% detection limit. Moreover, the AgNWs-based strain gauge demonstrated a reversible and reliable electrical response as a mechanical strain was periodically supplied at various frequencies. This simple, efficient and nonlithographic approach would be considered as an universal method to construct highly sensitive and reliable strain gauges for high resolution strain mapping fields.

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## 1. Introduction

High-sensitivity strain gauges are extremely essential to provide efficient structural health monitoring and detect the real-time activities of human organs, and promise abundant opportunities for wearable electronics [1–3], biomonitoring [4–6], and human/machine interface [7–9]. Currently, the conventional metal foil strain gauges cannot fulfill these demands as their gauge factor is typically in the range of 2–5, which is too low to detect the slight localized deformations. Compared to metallic foils, semiconductor strain gauges exhibit high sensitivity due to piezoresistive effects [7,10]. However, the drawbacks of semiconductor strain gauges, such as the small strain range, complex temperature compensation, narrow dynamic ranges and high fabrication cost are seriously limited their application in the next-generation highly sensitive strain gauges [11]. Therefore, it is fairly urgent to exploit a new category of highly sensitive strain gauge for effectively substituting the conventional metal foil and semiconductor strain gauges.

Recently, a great variety of nanostructure assemblies on the flexible substrates have been proposed as highly sensitive strain gauges, and metal nanoparticles [12,13], nanowires [14], carbon nanotubes [15], and graphene [16–18] have been widely evaluated for this purpose. Among these alternative materials, metal nanoparticles-based strain gauges, especially the strain gauges based on the assembly structures of metal nanoparticle, are more competitive and efficient than the other alternative candidates due to their high sensitivity, rapid temporal response and superior flexibility. These unique performance are attributed to the exponential dependence of the tunneling current on the interparticle distance and the amount of nanoscale gaps in the assembly structures [19]. The gauge factors of such nanoparticle-based strain gauges compare favorably to the commercial metallic strain gauges and are equivalent to the state-of-the-art semiconductor gauges [20].

To date, a number of approaches, such as convective self-assembly [20–22], layer by layer [23], stamping methods [24], chemical vapor deposition [11], drop casting [6,25] and electrodeposition [26], have been employed to fabricate such nanoparticles-based strain gauges on the flexible substrates. Although the strain gauges fabricated by these approaches exhibit high sensitivity and superior flexibility, these methods involve high-cost equipment and require high energy or complex operation procedures [27]. Moreover, the response speed and reliability

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of these strain gauges are not facile to regulate, which is extremely pivotal to efficiently monitor the structural health status in many engineering fields [28]. At present, it still remains a big challenge to explore an effective and facile strategy to construct the nanoparticles-based strain gauges in order to achieve highly sensitive and reliable detection.

As a facile and efficient patterning approach, ink-jet printing technique is considered as an excellent approach to organize the nanoparticles into the desirable assembly structures on various flexible substrates [29–32]. Compared with the aforementioned methods, it enables design flexibility and could assemble metal nanoparticles into multifarious patterns in an energy-conserving and nonlithographic manner [33–35]. Zhang developed a low-cost fabrication strategy to efficiently construct highly sensitive graphite-based strain sensors by pencil-trace drawn on flexible printing papers, and the strain sensors could be applied to variously monitoring microstructural changes and human motions with fast response/relaxation times [36]. Lewis reported a new method to fabricate highly stretchable sensors based on embedded 3D printing of a carbon-based resistive ink within an elastomeric matrix, and these strain sensors allows for creating soft functional devices for wearable electronics, human/machine interfaces by controlling print path and filament cross-section, respectively [37]. These methods provide a good opportunity to construct the nanoparticles-based strain gauges with high sensitivity and excellent reliability in a facile and efficient manner.

Following our previous study on AgNPs synthesis, assembly and ink-jet printed electronics [38–40], we here explored an efficient and facile strategy to fabricate a highly sensitive and reliable AgNWs-based strain gauges by regulating the coffee-ring effect with ink-jet printing technique. Taking advantage of this approach, a series of AgNWs with 5–6  $\mu\text{m}$  line width were successfully fabricated on the flexible PET substrate, and exhibited high sensitivity, temporal response, and excellent reliability as the active sensitive medium in the strain gauges. These prominent characteristics allow this nonlithographic approach as an universal strategy to construct various high-performance stain gauges in an efficient manner, and would have great potential for detecting real-time activities of human organs and routine local deformation of large-scale building structure.

## 2. Experimental

### 2.1. Chemicals and materials

Silver nitrate ( $\text{AgNO}_3$ ), ethylene glycol (EG) and polyvinylpyrrolidone (PVP,  $M_w = 1 \times 10^4$ ) were supplied by American Sigma-Aldrich CO., LTD. Ethanol and acetone were purchased from Sinopharm Chemical Reagent Beijing Co., Ltd. All of these chemicals were used as received without further purification. The other chemicals and materials used in the experiments were analytical or high-reagent grade. The water used in the experiments was ultrapure water (18.2  $M\Omega$ ) produced by a Milli-Q system.

### 2.2. Synthesis of AgNPs in solution-phase polyol route

As an effective approach, a solution-phase polyol route was adopted to synthesize the AgNPs with controllable shape and size distribution according to the literature [41]. In a typical synthesis procedure, PVP (3.6 g) was dissolved in ethylene glycol (30 mL), and  $\text{AgNO}_3$  (0.6 g) was added into the solution. The concentration of  $\text{AgNO}_3$  was approximately 1.6% by weight in the solution. The suspension was continuously stirred at 60 °C until  $\text{AgNO}_3$  was completely dissolved. Subsequently, the solution was slowly heated

up to 120 °C and remained for 1 h at this temperature to allow the reaction to complete. After ethanol and acetone were added into the reaction system, the AgNPs were easily separated by centrifugation at 8000 rpm. The AgNPs were dried under vacuum at 60 °C for 30 min before the further experiments. The UV–vis spectra, XRD spectra, and TEM and SEM images of the obtained AgNPs (Fig. S1) indicated that this method could synthesize AgNPs with  $30.2 \pm 4.6$  nm size distribution in high yield.

### 2.3. Controllable AgNWs assembly on the PET substrate induced by coffee-ring effect

The AgNWs on flexible PET substrate were assembled induced by the coffee-ring effect with ink-jet printing technique. Before ink-jet printing process, the obtained AgNPs were redispersed into a mixture of ethylene glycol and water by ultrasonication and ball milling. The concentration of AgNPs was approximately 0.5–2% by weight in the corresponding suspension. The obtained AgNPs ink was filtered through a 0.45 mm syringe filter and filled into the ink cartridge to fabricate the AgNWs assembly on the flexible PET substrate.

According to the design, the printing of the AgNPs ink was performed on a Dimatix Fujifilm DMP-2831 printer with 10  $\mu\text{L}$  Dimatix materials cartridge controlled by the Dimatix Drop Manager software. In the ink-jet printing experiments, only one nozzle was used in order to better control the AgNPs assembly morphology. The substrate temperature was set to 30 °C, and the humidity within the printing chamber was 30–40% RH. A series of AgNWs were fabricated by ink-jet printing the AgNPs ink onto the PET substrate, and the drop space was set as 30–50  $\mu\text{m}$ . The formed AgNWs on the PET substrate were sufficiently dried in a convection oven before the further experiments.

### 2.4. Fabrication of the AgNWs-based strain gauges

The strain gauges were fabricated by connecting the AgNWs with two 150 nm thick gold electrodes separated by distance 120  $\mu\text{m}$ . The stencil lithography was employed to deposit gold electrodes in order to avoid any contamination/degradation of the AgNWs on the PET substrate. To ensure the reliability and robustness, several AgNWs were simultaneously assembled on the PET substrate and connected by the deposited gold electrodes to construct the AgNWs-based strain gauges.

### 2.5. Electromechanical test of the AgNWs-based strain gauge

Prior to testing the response of AgNWs-based gauge to the applied mechanical strain, the electrical transportation in the quasi-static state was firstly characterized and the initial electrical resistances  $R_0$  were measured. The performances of fabricated AgNWs-based strain gauge were evaluated by monitoring the variation of their electrical resistance during the bending of the flexible PET substrate. The compressive and tensile strains were performed by bending them respectively. During the bending of the strain gauge, the electrical current was monitored with a voltage of 2.5 V by using a Keithley 4200-SCS semiconductor characterization system. The electrical resistance was calculated with the equation of  $R = V/I$ , where,  $V$  was the applied voltage, and  $I$  was the measured current, and the electrical signal was collected at 200 MSa/s. The horizontal coordinate of the mobile end and the vertical coordinate of nanoparticle assembly were recorded by digital laser rangefinder. These two coordinates were utilized to calculate the mechanical strain  $\epsilon$ , assuming that the geometrical profile of the PET film could be described as a circular with changing diameters. The strain  $\epsilon$  induced in the nanoparticle wires was estimated using

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