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A facile microwave approach to the fast-and-direct production of silver nano-ink

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ABSTRACT

In this work, we proposed a microwave method to the fast-and-direct preparation of silver nano-ink. The conductive ink is composed of multi-scaled silver nanoparticles (AgNPs) and a few of silver nanorods (AgNRs) as the conductive components. The differently sized nano-silver can provide high conductivity of the written tracks by affording high packing density. The existed one dimensional nanorods can build bridges between nanoparticles to decrease the silver content while maintaining the conductivity of the written pattern. The as-prepared conductive ink can be stored for 20 days without affecting its usability. The written tracks showed satisfying conductivity with a relatively low silver content of 0.025 mol L^{-1}.

1. Introduction

Printed electronics have provided a decent alternative to the conventional photolithography technology in the microelectronics industry. The photolithography process is a complex procedure that can cause serious materials waste and environmental pollution [1,2]. Printing technology is in essence a deposition process to transport the ink to the substrate. It expanded the choice range of the substrate materials, like paper. Paper substrate is inexpensive, biodegradable, lightweight, and can be folded into 3D configurations, which is in consist with the developing trend of electronics [3,4]. Conductive ink is the key among the three aspects of printer, conductive ink and substrate in printing technology. Due to the compatibility with high conductivity and low temperature processing, nano-metal conductive inks present a promising candidate for paper-based flexible electronic applications [5,6]. Among the numerous types of nano-metals, nano-silver has been explored most extensively because of its excellent electrical conductivity (σ_{Ag}=6.3×10^7 S m^{-1}) and oxidation stability [7,8].

There are two issues that currently prohibit nano-silver ink from large scale application: the inherent conflict between the solid content and the stability of the ink, and the ink preparation process is always complicated. For the commonly reported silver nanoparticles (AgNPs) ink, low solid content would decrease the thickness of the tracks, leading to high resistance of the pattern. Stabilizers like polymers are always applied to improve the stability of the conductive ink with high solid content [9–12], which would bring about trouble of removing the polymers in the deposited patterns. One feasible approach is to establish more conductive pathways under low content of conductive fillers [13]. In this paper, multi-scaled AgNPs and silver nanorods (AgNRs) were introduced as the conductive components, aiming at reducing the silver content by forming high compact density and "nanorods bridge" between separated AgNPs.

Regarding to the preparation method, most research relied on the typical three-steps process: synthesis of the nano-silvers, collection of them (often by centrifugation), and re-dispersion of the nano-silvers. It is difficult to avoid the collection process to obtain high solid content, resulting in the preparation process complicated and time-consuming. In the present paper, we introduced a facile and fast microwave approach to the preparation of nano-silver conductive ink. The coexistence of multi-scaled AgNPs and AgNRs in the ink was expected to provide high conductivity with low silver content.

2. Experimental section

2.1. Materials

Silver nitrate (Sinopharm Chemical Reagent Co. Ltd., GR), Polyvinylpyrrolidone (PVP K-30, Sinopharm Chemical Reagent Co. Ltd, GR), ethylene glycol (EG, Jiangsu Qiangsheng Chemical Co., Ltd., AR), Microwave oven (Glanz, G70F20CN3P-ZS (W0)).

2.2. Preparation of nano-silver conductive ink

0.17 g of silver nitrate and 0.34 g of PVP were added to 40 ml of ethylene glycol. The mixture was magnetically stirred until the silver

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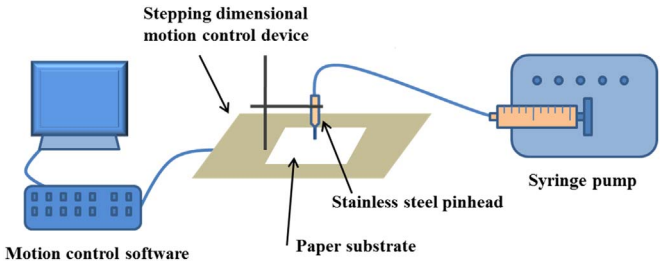

Scheme 1. Schematic of the direct writing system.

nitrate and PVP were completely dissolved. The obtained precursor was transferred to a container (100 ml volume) and put in the microwave oven. The reaction was carried out at 700 W for 90 s. The conductive ink was obtained after the boiling mixer cooled down naturally.

2.3. Direct-writing of silver pattern

In order to write patterns on paper substrate, a writing system consists of a stepping dimensional numerical control device and a syringe pump was developed (Schematic 1). The as-prepared conductive ink was filled in the syringe barrel connected to the dispenser with stainless steel pinhead. The flow rate of the ink was adjusted at 3 ml/h. The conductive ink was dispensed on the Epson photo paper to form desired tracks through running the G-codes in LabView.

To enhance the conductivity of the written pattern, the hot/pressure sintering method was applied. The hot/pressure sintering was implemented in a modified device by embedding a temperature control device to the powder tableting machine (Tianjin Bojun Science and Technology Ltd, BJ-30). Both sides of the paper were covered by thermal deformation resistant polyimide films to prevent the sample from contamination.

2.4. Characterizations

The surface morphologies of the written pattern before and after sintering were observed by scanning electron microscopy (S-4800 FESEM, Hitachi, Japan). The crystalline structure of the prepared nano-silver was examined by X-ray diffraction meter (XRD, DX-2600 with Cu Kα radiation). The breadth, length and thickness of the written pattern were measured by a metallographic microscope (DMI3000M/DFC 450, Leica), and the resistivity was determined by the 4-point probe method on a SM-4 system (Semishare) to calculate the electrical resistivity.

3. Results and discussion

Fig. 1(a) gives the SEM image of the obtained nano-silver, most of which shows spherical shape with a handful of rod-like ones. The corresponding size distribution result was displayed in Fig. 1(b), which showed a wide distribution range from 21 nm to 231 nm with a mean diameter of 86.43 nm. It is interesting to find that there was a proportion of 30% for particles from 105 nm to 231 nm, with the remaining 70% of particles sized in the range from 20 nm to 105 nm. According to Furnsa's random packing model [14,15], mixtures of powders with different sizes can give improved packing densities. The as-prepared multi-scaled nano-silver was expected to have a higher packing density than that of uniformed particles in 86.43 nm.

XRD measurement was carried out to verify the conductive component of the prepared nano-ink. As shown in Fig. 1(c), the significant diffraction peaks at 2-theta angle of 38.12°, 44.36°, 64.46° and 77.36° can be assigned to the (111), (200), (220), and (311) planes of face-centered-cubic (fcc) silver (JCPDS file No. 04-0783). The XRD

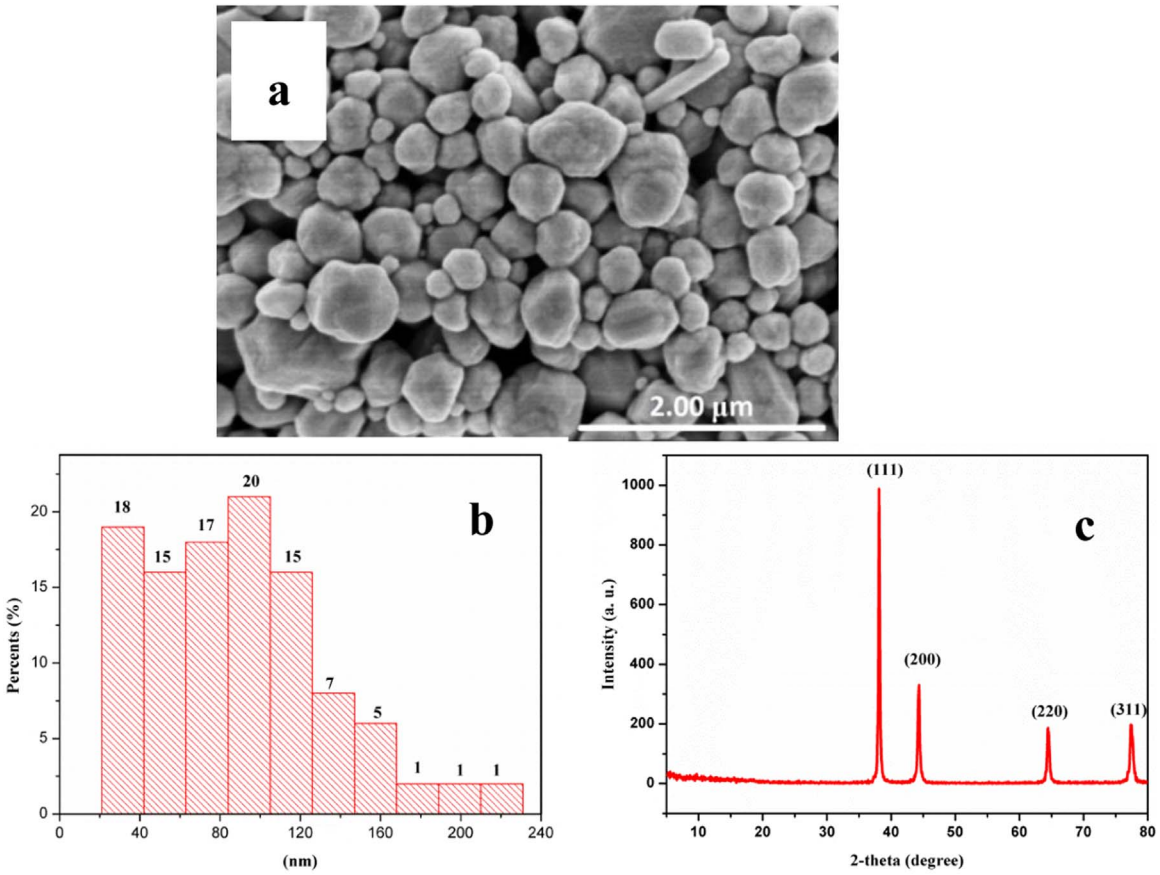

Fig. 1. (a) SEM image of the prepared multi-scaled nano-silver, (b) the particle sizes distribution and column bar, (c) the XRD pattern of the prepared nano-silver.