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Annealed graphene sheets decorated with silver nanoparticles for inkjet printing

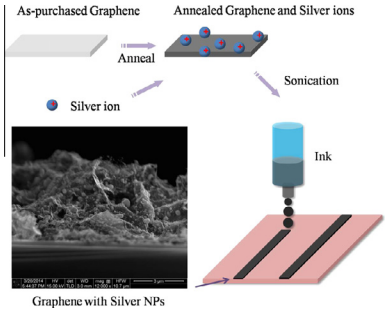
Gongkai Wang [a], Zhuo Wang [a,d], Zhihong Liu [b], Jiachen Xue [b], Guoqing Xin [c], Qingkai Yu [b], Jie Lian [c], Maggie Yihong Chen [a,*]

[a] Ingram School of Engineering, Texas State University, San Marcos, TX 78666, USA
[b] Ingram School of Engineering, and Materials Science, Engineering and Commercialization, Texas State University, San Marcos, TX 78666, USA
[c] Department of Mechanical, Aerospace & Nuclear Engineering, Rensselaer Polytechnic Institute, Troy, NY 12180, USA
[d] School of Material Science and Engineering, Chang'an University, 65 North Chang' an Road, Xi'an 710061, PR China

HIGHLIGHTS

- Annealed graphene nanosheets coupled with Ag organic complex ink is prepared.
- The microstructural changes of graphene nanosheets after annealing are confirmed.
- The resistivity of $4.62 \times 10^{-4}\ \Omega$ m of the track is achieved upon 15 printing times.
- The flexibility of the track on polyimide (PI) is measured.

GRAPHICAL ABSTRACT



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ABSTRACT

We report the preparation of annealed graphene nanosheets coupled with Ag organic complex inks by the approach of polymer stabilization and sonication, and the potential application of inkjet printing. The as-purchased graphene nanosheets were annealed as the main printing component which significantly increased the electrical conductivity of the inkjet printed track due to the highly reconstructed and nearly defect-free in-plane structures. Ag nanopaticles (NPs) were uniformly distributed on graphene nanosheets after post annealing process, which acted as superiorly conductive bridge to further decrease the contact resistance of graphene flakes and the resistivity of the inkjet printed track. The resistivity of $4.62 \times 10^{-4}\ \Omega$ m (conductivity of 2.16×10^{3} S/m) upon 15 printing times were achieved. The interwoven structured graphene nanosheets with Ag NPs formed a three-dimensional conductive network film with good mechanical robustness. The experimental results demonstrated the strategy of combining graphene nanosheets and nano-sized metal particles, which holds a possible pathway for the future application of flexible and maybe bendable inkjet printed electronic devices.

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1. Introduction

Electronics including touch screens [1], sensors [2], field-effect transistors (FETs) [3], light-emitting-diodes (LEDs) [4], photovoltaic cells [5], and electrochemical energy storage systems [6,7] coupled with features of multifunctionality, flexibility, stretchability, wearability, biocompatibility [8–13] is a rapidly evolving field, which has attracted extensive interests and efforts. Conventionally, the approaches of vacuum deposition and photolithographic patterning were well employed to fabricate electronics on flexible substrates. However, the limitations of chemical incompatibility in regarding

* Corresponding author. Tel.: +1 5122454158.
E-mail address: yc12@txstate.edu (M.Y. Chen).

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to flexible substrate materials and complex fabricating procedures during the integrated process partially retard the development of flexible electronics [14]. As a versatile technique, inkjet printing is under intense scrutiny for decades. The state-of-art inkjet printing offers a high efficiency, low cost, digital, non-contact, maskless and high resolution patterning process, holding a promising pathway to facilitate the development of flexible electronics in large scale [15–19].

Graphene could be a judicious selection as of primary importance component in inkjet printing for flexible electronics owing to its intrinsically high conductivity, flexibly mechanical robustness and chemical stability [20,21]. To date, graphene nanosheets could be produced by means of reduction from graphite oxide (GO) at relatively low expense in large scale [22]. However, the superior properties of graphene nanosheets were restricted at some extent by oxygen-containing functional groups generated during the oxidation process [23]. Limited conductivity (~15 S/cm) of inkjet printed films was obtained by using this kind of pristine reduction of GO (rGO) [24–26]. Accordingly, graphene-based ink still has to be tailored to be a contender for printed flexible electronics. Generally, annealing was a viable route to improve the quality of rGO nanosheets. The residual oxygen-containing functional groups anchored to the rGO basal plane after reduction could be removed at large extent along with the restoration of the π-conjugated structure, increasing the conductivity of rGO nanosheets [27,28]. Additionally, pure nano-size metal ink such as silver and gold also provides an alternative to conventional carbon-based ink [29–31]. Although the inherently very low resistivity makes itself feasible for inkjet printed flexible electronics, the cost efficiency and metal migration problems still quest for further exploration [25,32].

Whereupon, the strategy employed in this study was the combination of annealed graphene nanosheets and nano-size metal particles. Several factors either from annealed graphene nanosheets or nano-size metal particles conspired to improve the conductivity of inkjet printed track and lower the consumption of expensive metal materials. Specifically, as-purchased graphene powder was annealed as the primary component of the ink, aiming to improve the electrical properties without changing intrinsic structures itself. Stabilizing polymer was used to suspend annealed graphene nanosheets and tailor the viscosity of the ink for inkjet printing. The silver/organic complex reduced into silver nanoparticles as highly conductive factors. This further improved the conductivity of the printed track. Meanwhile, graphene nanosheets formed a flake to flake interconnected film, maintaining the mechanically robust feature of the printed track.

The morphology and microstructure of the graphene nanosheets and the inkjet printed tracks were evaluated using various characterization approaches. The post-annealing treatment of the inkjet printed tracks was optimized and the sheet resistance was investigated by the four-probe method. Additionally, the flexibility of the inkjet printed tracks on polyimide (PI) was measured.

2. Materials and methods

2.1. Materials

As-purchased graphene nanosheets (1–5 atomic layers, size of 0.5–5 μm) were received from Shanghai SIMBATT Energy Technology Co., Ltd., China. Ethyl cellulose (EC) (cat# 200646), Cyclohexanone (cat# C102180) and Terpineol (cat# 86480) were purchased from Sigma–Aldrich, USA. Silver/organic complex (TEC-CO-001) was purchased from InkTec Co., Ltd., USA.

2.2. Annealing of as-purchased graphene nanosheets

Annealing of as-purchased graphene nanosheets were carried out by using an induction heating furnace. The as-purchased graphene nanosheets samples were put into a graphite crucible and heated up from room temperature to 2200 °C at a heating rate of 1000 °C/h under a flow of Argon (flow rate of 500 sccm). Then they were kept at 2200 °C for 30 min.

2.3. Preparation of graphene based silver/organic complex inks

A simple approach of sonication was employed to prepare inks. Specifically, the annealed graphene nanosheets/EC powder was dispersed in an 85:15 mixture of cyclohexanone and terpineol. It was followed by sonication (Bath cleaner, BRANSON 2510) for 6 h to obtain a homogeneous solution (solids concentration of 2.0 wt.%, ~1.7 mg/ml graphene nanosheets) [33]. Then the silver/organic complex was added into the as-prepared solution by a volume ratio of 1:10 under an ice bath sonication for an extra 1 h. The annealed graphene based silver/organic complex ink was ready to use after final vacuum filtration to remove any large size particles. For comparison, a unitary as-purchased graphene nanosheets and annealed graphene nanosheets were added to prepare controlled ink samples respectively following the same procedures without the addition of the silver/organic complex. The concentration of the annealed graphene/Ag NPs ink was calculated and controlled as around ~1.7 mg/ml, which was comparable with that of pure annealed graphene. The solid content of the Ag NPs in the ink was about 0.02 wt.% calculated by the original mass of each component.

2.4. Inkjet printing

The Inkjet printing was carried out by using Fujifilm Dimatix Materials Printer (DMP-2800) equipped with a 10 pL drop cartridge (DMC-11610). The spherical status of ink droplet sequenced for printing was tailored by adjusting jetting voltage, frequency and viscosity wave form. The as-prepared inks were printed on Si/SiO_2 wafers at room temperature for general characterizations. The Si/SiO_2 wafers were sonicated in ethanol for 30 min followed by drying under a stream of Argon before printing. The as-prepared annealed graphene coupled with Ag/organic complex inks were also printed on PI at room temperature for flexibility measurement. All the inkjet printed samples were baked on VWR hotplate at ambient condition at 300 °C for 40 min before further characterizations.

2.5. Characterizations

X-ray photoelectron spectroscopy (XPS) was recorded using a PHI 5000 Versa Probe system. Raman spectroscopy was carried out with a Horiba Jobin Yvon Xplora confocal Raman microscope using a 532 nm laser as the probing light source. The X-ray diffraction (XRD) was performed on a Bruker D-8 X-ray diffraction system with a source wavelength of 1.542 Å at room temperature. Thermal gravimetric analysis (TGA) was carried out using a TA Instruments TGA-Q50 at the heating rate of 5 °C min^{-1} from room temperature to 600 °C in air condition. The morphology, microstructure and thickness of the inkjet printing samples were determined by a field-emission scanning electron microscope (SEM) on a Nova, NanoSEM, FEI. The sheet resistance of the inkjet printing samples was measured by a four-probe method using an appropriate geometry factor. The resistance under bendable condition was measured by a (FLUKE 8808A 5-1/2 DIGIT) multimeter performed on a home-made bendable system.

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