

Green synthesis of silver-graphene nanocomposite-based transparent conducting film



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ARTICLE INFO

Keywords:

Graphene
Silver
Nanocomposite
Reducing agent
Sheet resistance
Transmittance

ABSTRACT

In the present work, silver nanoparticles (Ag NPs)/graphene nanocomposite has been synthesized successfully by simple solvothermal method via green route. Citric acid is used as green reducing agent for the reduction of graphene oxide (GO) and Ag ions. Silver nitrate is used as a precursor material for Ag NPs. As synthesized Ag NPs/graphene nanocomposite has been characterized by X-ray diffraction, Raman spectroscopy, Fourier transform infra-red spectroscopy, UV–vis spectroscopy, thermal gravimetric analysis, field emission scanning electron microscopy, and X-ray photoelectron spectroscopy. Experimental results confirm the reduction of GO and the successful formation of Ag NPs decorated graphene nanosheets. In addition, spray coating technique is employed for the fabrication of transparent conducting films. Enhancement in the optoelectrical signatures has been achieved using thermal graphitization of fabricated films. Thermal graphitization at 800 °C for 1 h marks the best performance of fabricated film with sheet resistance of $\sim 3.4 \text{ k}\Omega/\square$ and transmittance (550 nm) of $\sim 66.40\%$, respectively.

1. Introduction

Transparent conducting films (TCFs) are an essential component of modern optoelectronic devices such as touch screen panels (TSPs), liquid crystal displays (LCDs), organic light-emitting diodes (OLEDs), and organic solar cells (OSCs) [1]. Indium tin oxide (ITO) is commercial material for TCFs having sheet resistance less than $100 \Omega/\square$ along with high transparency $\sim 90\%$ (at 550 nm wavelength). ITO shows DC conductivity to optical conductivity (σ_{DC}/σ_{OP}) ratio more than 35, and frequently utilized in the above mentioned applications. However, alternative materials (e.g. doped metal oxides, single-walled carbon nanotubes (CNTs), metal nanowires (NWs), printable metal grids and nanocomposites, etc.) have been searched extensively to fulfill the huge commercial demand due to the less availability of indium, high brittleness and high refractive index of ITO film [2,3]. Since the discovery, graphene: a one atom thick planar sheet of sp^2 hybridized carbon atoms in hexagonal lattice, has attracted scientific community worldwide by owing extraordinary properties including high optical transparency $\sim 97.7\%$, high room temperature mobility $\sim 2 \times 10^5 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, high Young's modulus $\sim 1 \text{ TPa}$, excellent fracture strength $\sim 130 \text{ GPa}$, and enormous thermal conductivity $\sim 5000 \text{ W m}^{-1} \text{ K}^{-1}$. Thus, it is a wonder material for variety of applica-

tions in the modern optoelectronic devices [4–6]. So far, various methods have been adopted for the synthesis of graphene nanosheets (GNs) such as chemical vapor deposition (CVD), arc discharge, and wet chemical routes, etc. [7,8]. Among these methods, wet chemical routes are more versatile and cost-effective way to produce GNs at large scale, followed by chemical reduction of graphene oxide (GO) in the presence of reducing agents (e.g. hydrazine and its derivative, etc.). These reducing agents are toxic/explosive in nature and have long lasting environmental impacts. Hence, green methodologies are preferable to produce GNs during reduction of GO. In contrast, restacking of graphene layers due to the van der Waals interaction becomes main drawback of wet chemical routes, suppress inherent properties of GNs, and limits their applications. Moreover, combination of GNs with different nanomaterials (e.g. nano metal oxide, nanorods, and metal grids, etc.) provide surface modification as well as restricts restacking of graphene layers [9]. Therefore, silver nanostructures (Ag Ns) combined with GNs have been explored for various applications including galvanic replacement reaction, optoelectrical properties, surface-enhanced Raman scattering (SERS), antibacterial activity and TCFs [10–12]. Particularly, TCFs based on Ag Ns/graphene (G) have been reported using various techniques by different groups. For example, Lee et al. have fabricated silver nanowires (Ag NWs)/G based

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<http://dx.doi.org/10.1016/j.physe.2017.03.015>

Received 6 October 2016; Received in revised form 17 March 2017; Accepted 20 March 2017

Available online 22 March 2017

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TCFs, which exhibits optical transmittance (T) ~88.6% at 550 nm and sheet resistance (Rs) ~19.9 Ω/\square [13]. Whereas, Hsiao et al. have fabricated G/Ag NWs hybrid nanomaterial based TCFs with Rs ~71 Ω/\square and T ~85% [14]. Besides Ag NWs, silver nanoparticles (Ag NPs) are being used as top electrode in the modern optoelectronic devices mainly due to their high electrical conductivity combined with the excellent light-trapping and anti-reflecting properties. Thus, Ag NPs together with GNs as a hybrid composite film shows excellent optoelectrical signatures. For example, Zhou et al. have fabricated Ag NPs/G multilayer film by electrostatic self-assembly process, and found Rs ~97 $k\Omega/\square$, T ~86.3% [15]. However, rGO/Ag NPs film has been fabricated using high Ag (~0.6%) concentration by Zhou et al., and film shows Rs ~8.3 $k\Omega/\square$ and T~89.2%, respectively [16]. Moreover, different process have been explored to fabricate GNs based TCFs such as Langmuir–Blodgett deposition [17], transfer printing [18], spin coating [19], dip coating [20], vacuum filtration [21] and spray coating [22,23]. Most importantly, poor dispersion of rGO/GNs in aqueous medium limits their use in TCFs fabrication [24]. Organic solvents such as N-methyl-2-pyrrolidone (NMP), N,N-dimethylacetamide (DMA), and N,N-dimethylformamide (DMF), etc. create homogenous dispersion with rGO/GNs, and mostly contains monolayers of rGO/GNs. Such homogenous dispersion are preferred, and coated directly over transparent substrate to fabricate better quality TCFs [25]. Additionally, thermal graphitization increases the crosslinking between sp^2 C-C bonds of small grain size GNs, which enhances the optoelectrical properties of TCFs [26]. Among available techniques, spray coating is the most efficient way to produce uniform TCFs of small grain size GNs [27].

Herein, Ag NPs/G nanocomposite has been successfully synthesized by solvothermal method using citric acid as green reducing agent for GO and $AgNO_3$ as precursor material for Ag NPs. Experimental results reveal the removal of oxygen species from GO by citric acid, and confirms the formation of Ag NPs /G nanocomposites. In addition, TCFs of Ag NPs/G nanocomposite have been fabricated by spray coating technique. Measurements of optoelectrical signatures have been carried out after thermal graphitization of fabricated TCFs. The schematic of various steps involved in the fabrication process of Ag NPs/G based TCFs is shown in Fig. 1.

2. Experimental

2.1. Materials

Graphite powder (Loba Chemie Pvt. Ltd., Mumbai, India) was used as graphite source for synthesis of GO. In addition, sulfuric acid (H_2SO_4 , 98%), citric acid ($C_6H_8O_7$), hydrogen peroxide (H_2O_2 , 30%), silver nitrate ($AgNO_3$), potassium permanganate ($KMnO_4$), sodium nitrate ($NaNO_3$), and N,N-Dimethylformamide (C_3H_7NO) were received from Qualigens Fine Chemicals, Mumbai, India. All chemicals were analytical grade and used without further purification.

2.2. Preparation of Ag NPs/G nanocomposite

Firstly, GO was prepared by modified Hummer's method as reported elsewhere [28]. Then, as synthesized GO (500 mg) was mixed in the 100 ml of DI water, and sonicated for 3 h to get homogenous dispersion. Thereafter, 0.5 M $AgNO_3$ and 0.1 M $C_6H_8O_7$ were mixed into GO suspension, and stirred rigorously for 1 h. Further, formed blackish dispersion was transferred to 100 ml N_2 purge teflon lined autoclave at 120 °C for 3 h. Finally, obtained grayish solution was filtered by 0.2 μm membrane filter, and washed several times with DI water to remove residual impurities; and dry at 80 °C for overnight in vacuum oven. Final product was dry powder of Ag NPs /G nanocomposite. For comparison GNs have been synthesized in the same condition (GO 500 mg in the 100 ml DI with 0.1 M $C_6H_8O_7$).

2.3. Synthesis of thin nanocomposite film

Spray coating was used to deposit TCFs of Ag NPs/G nanocomposite [29]. Ag NPs/G nanocomposite (two samples having concentrations of 1 and 3 mg/ml) was mixed in 10 ml DMF, sonicated for 1 h, and stirred for 8 h at 80 °C. The resultant dispersion was allowed to cool at room temperature, and centrifuged at 3000 rpm for 0.25 h. Then, top 5 ml of Ag NPs/G/DMF dispersion was sprayed onto a 25×25 mm² preheated quartz substrates and named as AGF-1, AGF-2 for 1 and 3 mg/ml, respectively. Before coating, quartz substrates were ultrasonically cleaned with DI water, acetone subsequently and dried in vacuum oven. Then, quartz substrates dipped into piranha solution

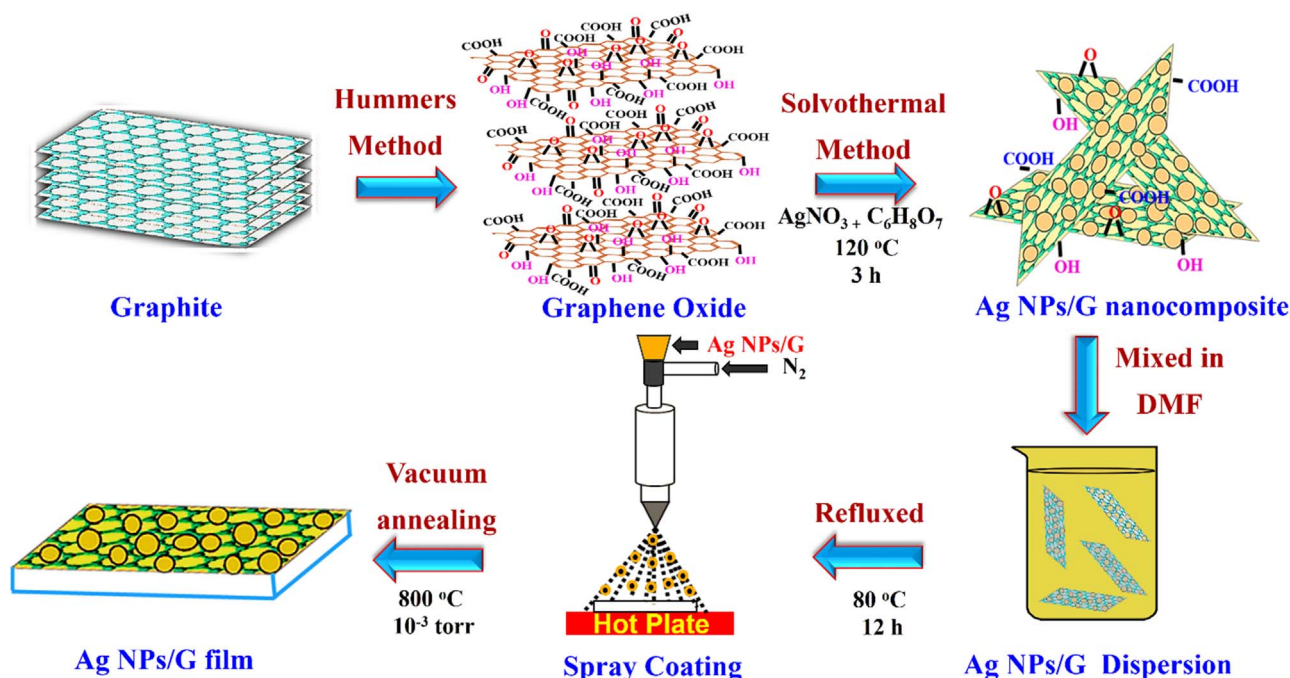


Fig. 1. Schematic of various steps involved in the fabrication process of Ag NPs/G based TCFs.

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