



Preparation of self-supporting graphene on flexible graphite sheet and electrodeposition of polyaniline for supercapacitor



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ABSTRACT

A graphene-based composite was prepared by the in-situ growth of a self-supporting graphene (SSG) on a flexible graphite sheet (FGS) via electrochemical intercalation of FGS and then the electrodeposition of a thin, homogeneous, and conformal polyaniline (PANI) film. The maximum areal capacitance of 1.36 F cm^{-2} and specific capacitance based on PANI of 491.3 F g^{-1} were achieved for the prepared FGS-SSG/PANI composite. The good cycling stability of FGS-SSG/PANI was proved by cyclic voltammetry at a scan rate of 50 mV s^{-1} for 3000 cycles. The symmetric supercapacitor device assembled using FGS-SSG/PANI composite electrodes exhibited a high energy density of 46 W h kg^{-1} at a power density of 275 W kg^{-1} on the basis of the total mass of PANI ($\sim 10\%$ of the total mass of the flexible electrodes). The good electrochemical properties indicate that the FGS-SSG/PANI is a promising flexible electrode for supercapacitors.

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1. Introduction

Graphene, with a single layer or only a few atomic layers, is a two-dimensional honeycomb sp^2 -hybrid carbon material [1–3]. Considering its large specific surface area [4] and high electric conductivity [5], graphene is usually used as a supercapacitor electrode material. However, due to Van der Waals interactions [2,6] and π – π stacking [2], the irreversible aggregations and even stacking easily occur in the course of preparation, dispersion, drying, and further processing. Consequently, it is necessary to explore valid approaches to avoid or decrease bulk-quantity aggregations. Generally, the surface modification of graphene [7,8] and the introduction of the second component [9–11] are both strategies that can be used to inhibit aggregations.

Polyaniline (PANI) is an attractive supercapacitor electrode material due to its low cost, high capacitance, simple synthesis procedure, good environmental stability, and redox reversibility [12,13]. However, the main weakness of PANI is its poor electrical conductivity. A combination of PANI and carbon nanomaterials

allows high specific capacitance, excellent conductivity, and long cycling life. A number of studies on the synthesis of nanocarbon-PANI composites have been reported [14–19]. For example, Jiang et al. prepared electrochemically reduced graphene oxide-polyaniline composites in a two-electrode cell which displayed a large capacitance (195 – 243 F g^{-1}) at 100 mV s^{-1} and an excellent electrochemical stability [14]. Otrokhov et al. reported polyaniline/multi-walled carbon nanotubes composite with a core-shell structure [17]. The specific capacitance of this composite was 440 F g^{-1} at 5 mV s^{-1} and decreased by less than 7% after 1000 cycles.

However, a PANI layer on carbon substrates with complete coverage would separate conductive carbon connections as well as direct contact with the current collector in the electrode, and consequently increase the contact resistance. In order to construct efficient conducting channels, we design a graphene-PANI composite for supercapacitor by the electropolymerization of aniline monomer to form a conformal PANI film on a self-supporting graphene (SSG), which is grown in-situ on the surface of a flexible graphite sheet (FGS) via electrochemical exfoliation. FGS is an industrial production prepared through rolling exfoliated graphite flakes, which are obtained by the intercalation of anions into graphene layers of natural graphite and then exfoliation under

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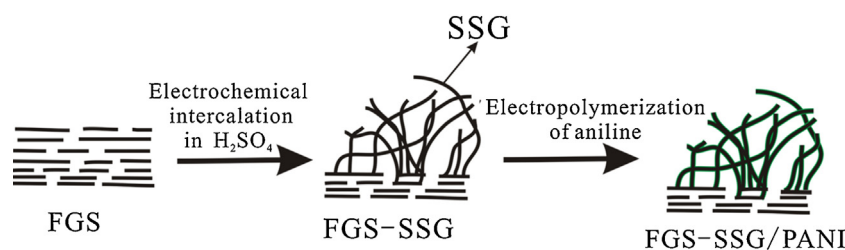


Fig. 1. Schematic cross-section of the electrochemical intercalation of FGS and the electropolymerization of aniline on SSG.

thermal treatment conditions [20]. During the process of FGS preparation, the attractive force between the graphene layers becomes weaker. It is expected that graphene is easily produced via the re-intercalation of FGS in which the exfoliated graphite flakes are only mechanically interlocked with each other. Moreover, FGS with high flexibility, conductivity, and chemical stability can be applied as a flexible electrode in supercapacitors, batteries, and fuel cells [20–23]. So we chose FGS as a flexible substrate to produce SSG in this study. The schematic cross-section of electrochemical intercalation for FGS is shown in Fig. 1. The process of intercalation is carried out in a solution containing sulfuric acid. Under electric field conditions, SO_4^{2-} anions intercalate into graphene layers of FGS, leading to the expansion and formation of graphene on the surface of FGS. The surface graphene bounding with the un-intercalated FGS substrate (defined as SSG) contributes a large surface area. To consolidate SSG and achieve a high capacitance, a thin PANI film is electro-deposited on the SSG. The obtained product can be used as an electrode without conductive agent or binder. The unique structure, in which FGS is used as a flexible current collector

directly and SSG is grown in-situ on the surface of FGS, provides conducting channels and ensures the maximization of the electrochemical utilization of the PANI pseudocapacitance.

2. Experimental

2.1. Preparation of FGS-SSG

FGS (Density: 1.0 g/cm^3 , $1 \text{ cm} \times 3 \text{ cm} \times 0.02 \text{ cm}$) was washed with anhydrous ethanol and distilled water. FGS-SSG was prepared as follows. First, a pair of FGSs was used as electrodes and placed in an electrochemical cell filled with the electrolyte of sulfuric acid solution with a pH value of 1.5. The exposed area of FGS below the liquid level was kept at 1 cm^2 ($1 \times 1 \text{ cm}$), and the rest of the electrode was sealed with insulating coating. The electrodes were placed in parallel with a separation of 1 cm. Afterwards, a constant potential of 8.0 V (DC voltage) was applied to two electrodes for 10 min. Finally, the anode was washed several times with distilled water, and dried at 80°C for 6 h in a vacuum oven. The resulting electrode was defined as FGS-SSG.

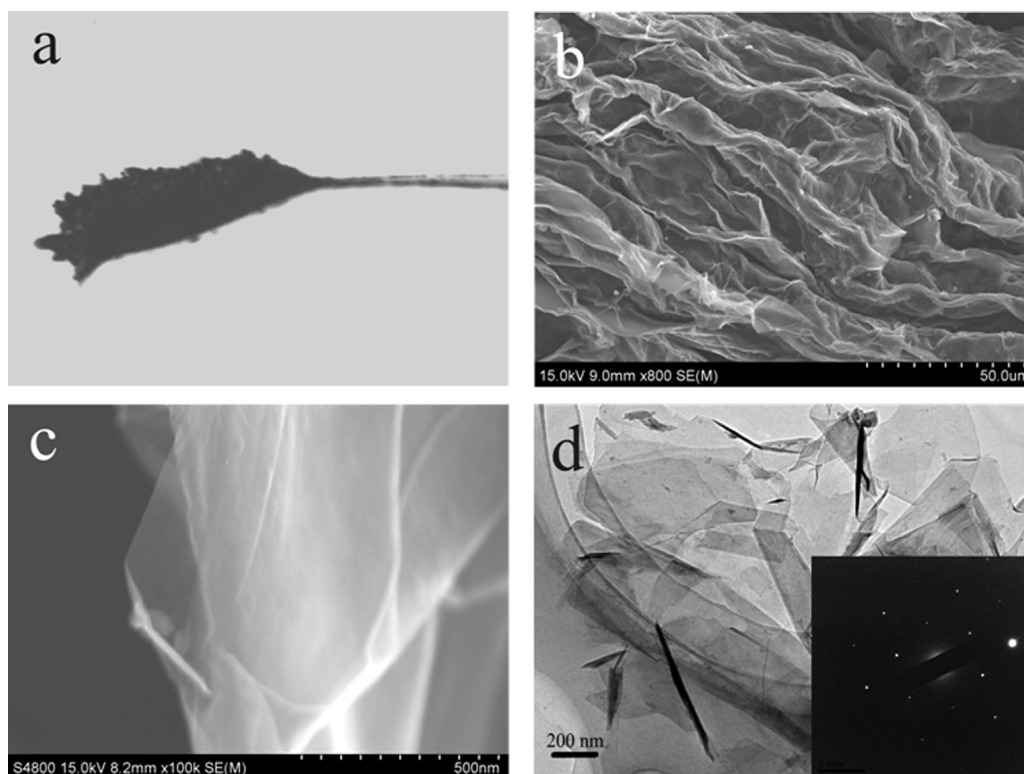


Fig. 2. (a) Digital photograph and (b) SEM image of the cross section for FGS after electrochemical intercalation. (c) SEM image of FGS after electrochemical intercalation. (d) TEM image of the black powders peeled from the surface of FGS after electrochemical intercalation. The inset of (d) is the corresponding SAED pattern.

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