



A graphite foil electrode covered with electrochemically exfoliated graphene nanosheets

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ABSTRACT

We demonstrate a highly efficient and large area synthesis of 2-D graphene nanosheets on the surface of flexible graphite foils by electrochemical exfoliation of graphite in an effective electrolyte, poly(sodium-4-styrenesulfonate) solution.

A constant current of 150 mA/cm was applied to the vertically aligned graphite (anode) and copper (cathode) sheet in the PSS electrolyte solution during a preset time for electrolytic surface exfoliation of the graphite sheet; uniform expansion of the graphite foil was observed. This expanded foil was characterized using scanning electron microscopy, confocal laser scanning microscopy, and high-resolution transmission electron microscopy. Furthermore, we demonstrate the ability of this high surface area foil, covered with uniform graphene, to enable improved electrolyte permeability and Li ion transfer, thereby enhancing electrochemical performance of Li ion battery electrodes.

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1. Introduction

Graphene, a two-dimensional (2-D) form of graphite, has attracted attention for applications in nanoelectronics, composites, molecular gas sensors, and field emission devices due to several fascinating properties that include atomic thickness/high surface area, high mobility of charge carriers, high mechanical strength, and excellent thermal conductivity [1,2]. Recently, graphene has been also considered as a good insertion host for high-capacity lithium ion battery electrodes [3–5].

In conventional lithium ion batteries, graphite and graphitized carbonaceous materials have been primarily used as anodes. A wide range of structures, and thus properties, is possible in this family, depending upon how the carbon is produced. For example, enhanced mechanical, thermal, and electrical properties of a flexible graphite sheet have made it a high-energy Li ion battery electrode. However, this compressed flexible graphite sheet exhibited comparatively low and highly irreversible capacities due to unfunctionalized layered graphitic structures, hindering permeability of the electrolyte and Li ion transfer [6].

Recently, simple and effective methods for the synthesis of freestanding exfoliated graphene sheets from graphite have been investigated using laser ablation, arc-discharge, and solvothermal/electrochemical processes [1,2,7,8]. More importantly, Wang et al. reported that chemically prepared graphene paper exhibits distin-

guishable electrochemical properties compared with graphite [9]. Herein, we demonstrate the formation of graphene directly onto the entire surface of flexible graphite foils using an electrochemical exfoliation process in an aqueous electrolyte solution containing poly(sodium-4-styrenesulfonate) (PSS). Furthermore, the enhanced specific capacities of these controlled high surface area graphite foil electrodes and their application as anodes in Li ion batteries are reported.

2. Experimental

The presented synthetic strategy to modify the surface of the graphite foil is illustrated in Fig. 1a. A convenient starting material for the stationary anode is a flexible graphite foil, Grafoil® (0.25 mm thick, GrafTech). A copper foil (25 μm thick, Aldrich) supported by a glass plate, was used as the counter electrode with a separation of 2 cm. For synthesis of the graphene on the graphite foil by electrolytic exfoliation, 0.001 M of an aqueous electrolyte containing PSS (molecular weight = 70,000, Aldrich) was used, similar to an electrochemical synthetic process of graphene reported [8]. The constant current between the two electrodes was set at 300 mA for duration from 1 min to 2 h, using a DC power supply (model E3612A, Agilent). Corrosion of the graphite foil could be clearly observed based on the expanded foil and appearance of black powder near the foil electrode during the electrolytic exfoliation process (Fig. 1b and c). Subsequently, the exfoliated graphite foils were washed with deionized water and ethanol, and then dried in a vacuum oven at 60 °C for 2 h.

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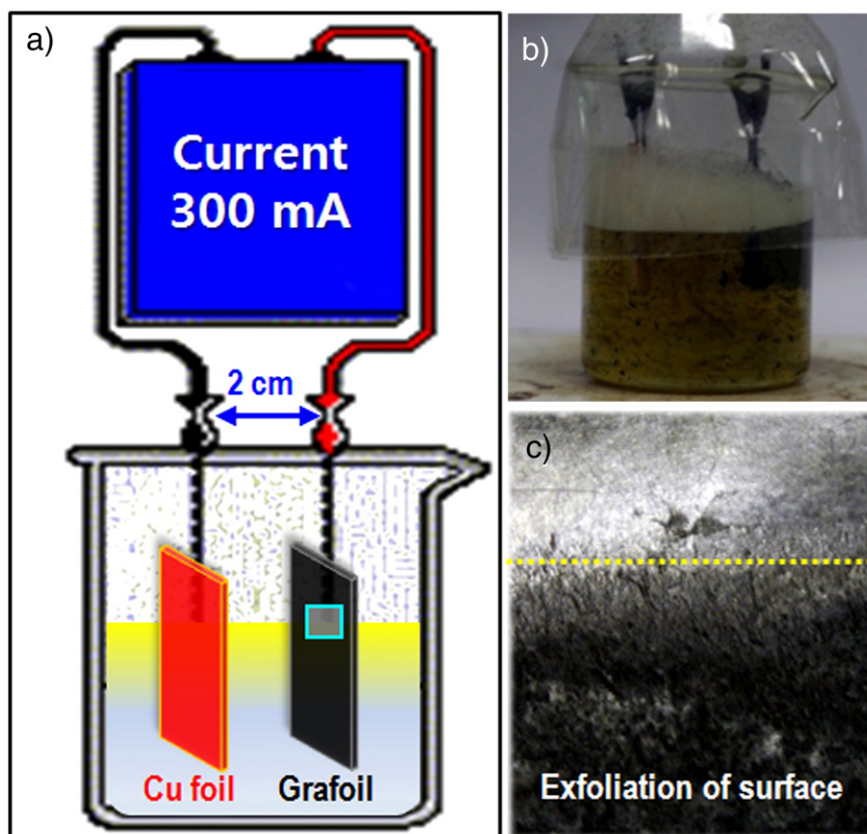


Fig. 1. (a) Schematic diagram and (b) digital camera image of experimental set-up. (c) Digital photograph for the surface of the graphite foil anode after electrolytic exfoliation.

The surface morphology of the exfoliated graphite foils was characterized by scanning electron microscopy (SEM, model JSM-6700F, JEOL), confocal laser scanning microscopy (model OLS-3000, Olympus), and high-resolution transmission electron microscopy (HRTEM, model JEM-2100F, JEOL).

Electrochemical tests were performed in Swagelok™-type cells assembled in an argon-filled glove box. The as-exfoliated graphite foils were directly used as the positive electrodes without any cohesive polymer binder or conductive additives. The mass of positive electrodes were around 13–17 mg but their geometrical surface areas were fixed to be 0.8 cm^2 . Each cell was composed of a positive electrode, a Li metal foil as a negative electrode, and a separator film (Celgard 2400) saturated with a liquid electrolyte consisting of 1.0 M LiPF_6 dissolved in ethylene carbonate and dimethyl carbonate (1:1 by

volume). The cyclic voltammetry (CV) measurements of the assembled cells were carried out at a scanning rate of 0.3 mV s^{-1} . The cells were also galvanostatically cycled between 0.0 and 2.0 V using a battery cycler (WBCS 3000, WonATech).

3. Results and discussion

Fig. 2a shows a low-magnification, plane-view, SEM image of the as-received graphite foil used in this study, which consisted of natural graphite flakes. This foil was shown to possess a smooth morphology. After a 1 h electrolytic exfoliation process, however, the surface microstructure was altered to a curled morphology, intrinsic to the graphene [3,4], consisting of a thin wrinkled paper-like structure (Fig. 2b). Indeed, the representative TEM and HRTEM images taken

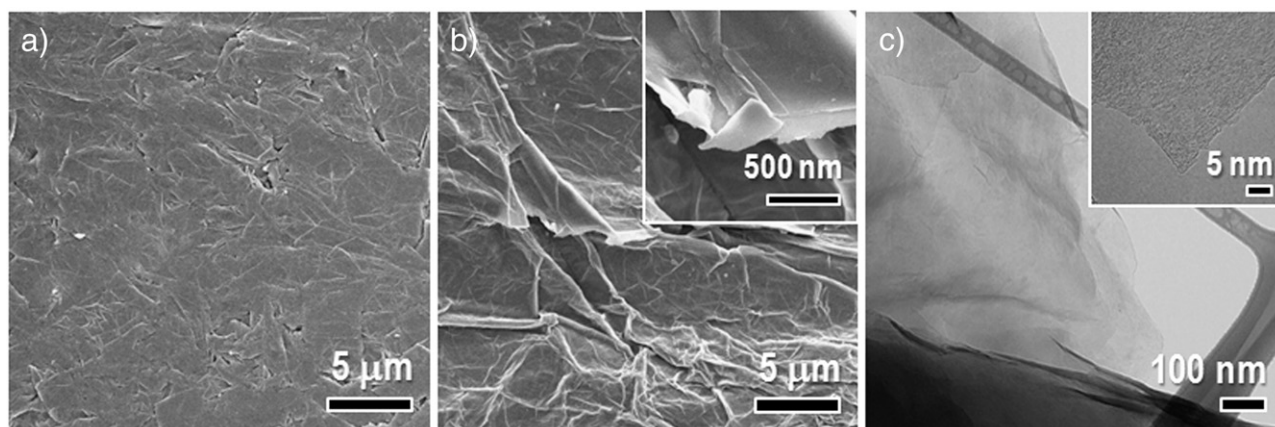


Fig. 2. (a) and (b) SEM images for the surface of as-received and exfoliated graphite foil after the electrolytic process (1 h), respectively. Inset shows magnified image. (c) TEM image for the black precipitate peel from the surface of the exfoliated graphite foil after the electrolytic process (1 h).

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