Developing Manufacturing Process for the Graphene Sensors

Abdullah Faqihi, John Hedley

Abstract—Biosensors play a significant role in the healthcare sectors, scientific and technological progress. Developing electrodes that are easy to manufacture and deliver better electrochemical performance is advantageous for diagnostics and biosensing. They can be implemented extensively in various analytical tasks such as drug discovery, food safety, medical diagnostics, process controls, security and defence, in addition to environmental monitoring. Development of biosensors aims to create high-performance electrochemical electrodes for diagnostics and biosensing. A biosensor is a device that inspects the biological and chemical reactions generated by the biological sample. A biosensor carries out biological detection via a linked transducer and transmits the biological response into an electrical signal; stability, selectivity, and sensitivity are the dynamic and static characteristics that affect and dictate the quality and performance of biosensors. In this research, a developed experimental study for laser scribing technique for graphene oxide inside a vacuum chamber for processing of graphene oxide is presented. The processing of graphene oxide (GO) was achieved using the laser scribing technique. The effect of the laser scribing on the reduction of GO was investigated under two conditions: atmosphere and vacuum. GO solvent was coated onto a LightScribe DVD. The laser scribing technique was applied to reduce GO layers to generate rGO. The micro-details for the morphological structures of rGO and GO were visualised using scanning electron microscopy (SEM) and Raman spectroscopy so that they could be examined. The first electrode was a traditional graphene-based electrode model, made under normal atmospheric conditions, whereas the second model was a developed graphene electrode fabricated under a vacuum state using a vacuum chamber. The purpose was to control the vacuum conditions, such as the air pressure and the temperature during the fabrication process. The parameters to be assessed include the layer thickness and the continuous environment. Results presented show high accuracy and repeatability achieving low cost productivity.

Keywords—Laser scribing, LightScribe DVD, graphene oxide, scanning electron microscopy.

I. Introduction

Biosensors play a significant role in the healthcare sectors scientific and technological progress. Clark was the first to present the concept of biosensors in 1962 and called them enzyme electrodes [3], [4]. Recently, biosensors have attracted more attention in the medical and nanotechnology sectors. Others have developed a multitude of biosensors for biotechnology, food and beverage productions, environmental, agricultural, and medical applications [8], [13]. For example, a sensor was developed based on the protein-based cancer biomarkers to detect biomolecules in order to identify the disease or the drug indicators while sensors in the medical field have been used to monitor vital signs, to diagnose, and to improve the critical care of patients. In recent years, the need for early detection and diagnosis of diseases has encouraged scientists to develop novel approaches in biosensing.

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There are now many different methods available for the manufacture of graphene. These methods produce graphene of varying quality, cost and size, which in turn make them suitable for different applications. Starting with the mechanical exfoliation, which producing excellent graphene samples, the chemical vapour deposition, thermal decomposition and reduction of graphene oxide, which considered to be the most popular method of producing graphene. Each method has its own features, drawbacks and limitations which vary, dependent upon its intended use, we refer the reader to [10], for more details.

Nowadays, many researches are investigated to study the biosensors. Studies on biosensors face several difficulties such as combining micro fluids with electronics and developing biological samples. It is, however, important to note that biosensors have various benefits. Not only because biosensors easy to use, cost-effective, and disposable, but they can also be measured in real time and are portable as they are small in size. In addition, they only need a small volume of sample while simultaneously providing a quick diagnosis [1], [2]. Graphenes innovative features has made it significantly advantageous for biosensors as well as chemical sensors [5], [11]. Further, graphene has single-atom thickness and considerable carrier mobility, thus offering remarkable sensitivity for graphene-based sensors while also presenting an exceedingly large surface/volume ratio [10].

Many experimental works have been presented to investigate the biosensors, for researchers in [12] used mechanical stripping to develop a stable monolayer of graphene at the University of Manchester. This helped address the problem that the international physics and material communities had been struggling with for approximately 50 years regarding whether graphene can be obtained experimentally or was a hypothetical structure. Graphene, as can be gathered from the name, is two-dimensional carbon nano-materials single atomic layer and is connected with the ubiquitous graphite has been investigated by researchers in [9]. It is noteworthy that the unprecedented characteristics of graphene as well as GO and rGO helped develop electrochemical biosensors for identifying biomolecules, such as electron transferability, chemical stability, large surface area, and economic feasibility [7]. Laser scribed graphene (LSG) is an innovative platform concerning patterned electrochemical biosensors. It is applicable to amperometric, chemiresistor, as well as impedance sensors as it is simple as well as has a low-cost manufacturing method. The LSG method involves the graphene oxide being converted into reduced graphene oxide using a laser. The LightScribe DVD drive can also be used to attain particular patterns.

In the current study, two types of graphene electrodes were manufactured using the technique described above, which is development of the techniques presented by researchers in [6]. This paper is organized as follows. Description of the experimental work and the developed technique is presented in Section II. In Section II-C, we present case study and example . Our approach is shown to enjoy the expected accuracy as well as the efficiency. Concluding remarks are given in Section IV.

II. Description of the Experimental Technique and Methodology

In this study a GO solvent was purchased from Graphenea SA, with the concentration of $0.4wt$ and $1.5g/cm3$, respectively. Transparent Polyethylene terephthalate (PET) foil with thickness of $100m$ was applied as a flexible substrate for GO. The manufacturing system passed through different stages, as in the following steps:

A. Preparation of the Sensor Device Using LSG Based on GO

In this stage, preparation of the sensor device using LSG based on GO DVD disc was used as the base substrate where the PET foil was glued on it using 3M spray to create the base layer. An amount of $15ml$ of GO solvent was homogeneously drop-casted over PET foil. The GO solvent was then left to dry for 48 hours at room temperature (25 C). Upon preparation of the substrate, electrodes were designed using the Autodesk software and loaded as the input onto the LightScribe Template Labeler Version 1.18.27.10 by Hewlett-Packard. Initially, a circular disk pattern was selected. The DVD disc was inserted bottom-up inside the LaserScribe writer and the selected design loaded from the computer. The disc was executed by the laser scriber for ten cycles, with each cycle taking 12-15 minutes, as shown in Fig. 1, in addition to the images for the final design configurations is depicted in Fig. 2, for more illustration.

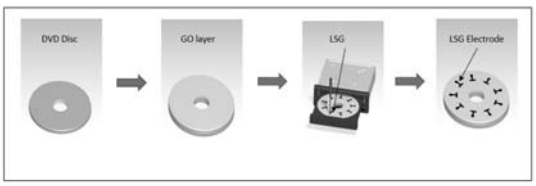

Fig. 1 The overall process of patterning LSG electrodes

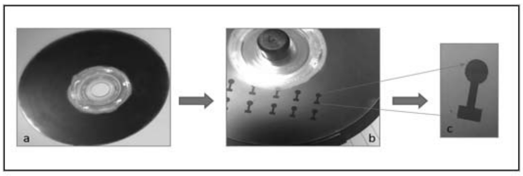

Fig. 2 Images of the final design for the first sensor: (a) GO coated substrate film stuck on a DVD disc; (b) Interdigitated capacitor sensors imprinted by LightScribe onto the GO; (c) Magnified image of an Interdigitated capacitor sensor formed by ten parallel electrodes

B. Preparation of Sensor Device Using LSG Based on GO with Vacuum CChamber

In this stage a stainless-steel vacuum chamber was assembled and used to draw comparison between the electrodes created in vacuum environment and room conditions shown in Fig. 3. It was also applied to improve and reduce the fabricated graphene (rGO) electrodes to obtain optimum physical properties. Such as conducting, flexible and reduced thermal conductivity.

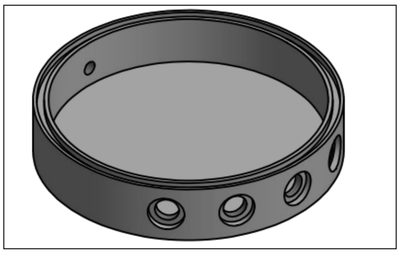

Fig. 3 Image of the vacuum chamber

Similar to the initial experiment, the disc was executed by the laser scriber for ten cycles inside the chamber at a pressure of $1.1 * 10^{-1}\ mbar$ to obtain the optimum properties of the sensor. Fig. 4 shows that some scratches on the surface of the disc resulted from the pressure.

Fig. 4 The experimental setup in the laboratory

C. Morphological Analysis of GO and rGO

In the study of topology and lateral dimensions, determining the thickness of sheets can be challenging. In the case of GO,

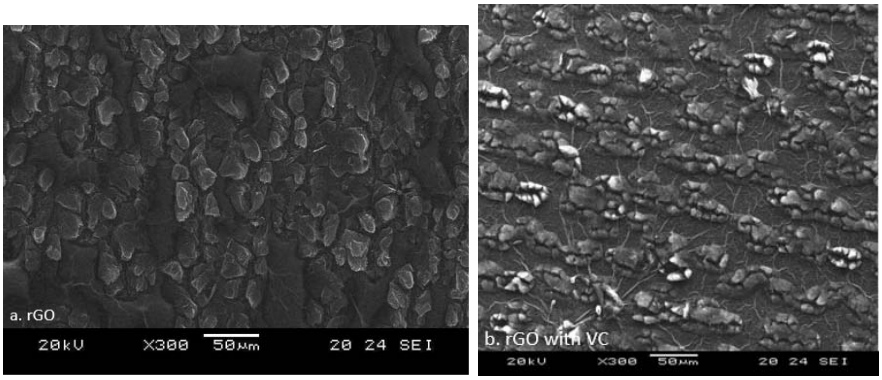

Fig. 5 Microstructural details (SEM) of rGO before and after vacuum chamber (VC)

it usually exhibits two-dimensional nanosheet morphologies with wrinkled texture, uneven margins and surface roughness due to scrolling [3]. Scanning electron microscopy (SEM) was used to examine the morphology of the GO and rGO composites under two conditions: room and vacuum conditions. Fig. 5 reveals the rGO composites which reflect a similar close-surface composition as a result of the laser scribe which causes the process of exfoliation of the GO layer.

III. Case Study

In this section the Raman spectra of the strips are produced by laser-scripting a CD coated with a GO solution by a DVD writer in air and depict intense D and G bands. A relatively flat 2D band was also observed but is not presented in the figure. The figure also shows that the G band occurred at a wavelength of 1587 cm-1, which is a shift from the usual graphite G band of 1570 cm-1. This was due to the oxygenation of the graphite. Meanwhile, the D band occurred at a wavelength of 1355 cm-1 due to defects, vacancies, and distortions of the SP2 domains after oxidation was completed. Both occurrences agree with literature, D at a wavelength of ca.1350 cm-1 and G at ca.1595 cm-1. The study of crystal structure for rGO electrodes made under the vacuum condition revealed both D (1355 cm-1) and G (1584 cm-1) bands similar to the wavelengths of atmospheric condition. Researchers in [6] reported a variation in intensity ratios at 0.9-1 and 0.7-1 for GO and rGO, respectively. Furthermore, they observed the intensity ratio for a thermally-reduced GO which was $ID/IG > 1$ for a reduction period of two hours. However, by increasing the period to two and five hours, the intensity became significantly smaller than that of GO, and a continued reduction in the value of intensity might indicate the graphitisation of the GO/r-GO. Hence, the value of intensity ratios in both cases was an indicative for the presence of rGO.

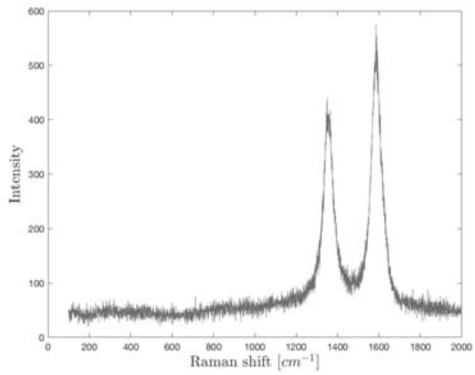

Fig. 6 The experimental outcome for the first sensor based on GO under vacuum chamber

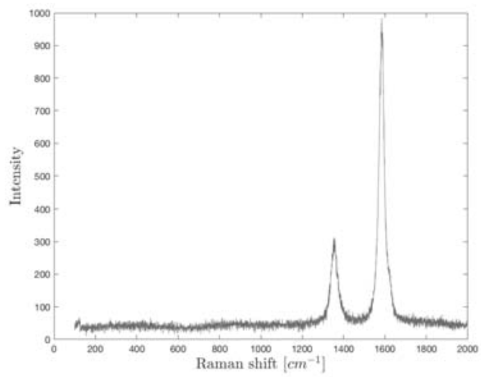

Fig. 7 The experimental outcome for the first sensor based on GO under vacuum chamber

IV. Conclusion and Future Work

This study produced LSG device models based on reduced graphene oxide (rGO) using laser scribing in air. A developed approach has been applied in this experimental study to manufacture and optimise conventional graphene electrodes by using the DVD scribing technique modified by way of scribing in a vacuum chamber. The results of Raman spectroscopy of the produced LSG in vacuum show that Id/Ig ratio was 0.3, while in air the ratio is 0.7 this shows a change of 57% after using the vacuum chamber method. Further studies should be undertaken in order to identify the physical and chemical properties of graphene electrodes, enabling these to be exploited in the fabrication of biosensing devices.

The optimisation of biosensor devices is a fruitful area for further study, particularly in terms of focusing on the acquisition of high-quality properties for the biosensor model. In this case, the parameters to be assessed include environmental pressure and layer thickness. Another promising area for future research relates to the use of X-ray photoelectric spectroscopy (XPS) to examine the surface chemistry structure of GO and rGO. Additionally, future researchers could use Raman spectroscopy to identify the quality of GO and rGO structures. Studying these parameters with varying pressure is promising future work and next step in this research. Finally, one possibility could be to use the produced sensing devices as biosensors in the measurement of biological samples utilising CV (Cyclic Voltammetry).

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