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A graphene-based sensor for real time monitoring of sun exposure

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A R T I C L E I N F O

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ABSTRACT

The photoreduction of graphene oxide (GO) with titanium dioxide (TiO₂) was exploited to fabricate a UV sensor for real time monitoring of sun exposure. The sensor was fabricated by simultaneous deposition of GO sheets and TiO₂ nanoparticles onto interdigitated electrodes using an AC electrophoresis deposition method. Changes in the resistance of the GO sheets decorated with TiO₂ nanoparticles during repeated cycles of exposure to UV were measured to understand the sensitivity of this sensor to UV radiation. Current-time traces revealed that the fabricated UV sensor retains a memory of each cycle of UV exposure; regardless of whether the sensor is exposed to UV for one long cycle or several short cycles. This memory of the extent of UV exposure is a biomimetic approach, analogous to the response of the skin to sun, and means the sensor requires no power except when the data is read from the sensor.

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1. Introduction

The sun's UV radiation is vital to vitamin D production but overexposure to UV radiation can cause sunburn [1]. Colour changeable wearable UV sensors can help us to avoid over exposure to the sun. One strategy for such sensors is to use halogenated organic materials that produce acidic compounds in response to UV radiation. This produces a change in colour of the top layer, which contains a pH indicator [2]. Another strategy by Armani et al. used the colour change of a polymer due to degradation by exposure to UV radiation [3]. A third example relevant to our work is gradual discoloration of a food dye by titanium dioxide (TiO₂) [4]. This sensor has an adjustable decolouration rate that can be tuned to different skin types or SPF-rating sunscreens [4].

To enable real time monitoring, some wearable sensors are connected to a smart phone via Bluetooth. These are mainly siliconbased sensors that provide relative UV intensity data [5]. Exposure is subsequently calculated by considering the measured UV index, and the user is notified *via* an alarm if safe exposure limits are

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exceeded. Therefore, having a wearable sensor that is selectively sensitive to UV radiation and memorizes all cycles of exposure to UV can be advantageous. Here, photo-induced reduction of graphene oxide to graphene by TiO_2 is exploited to achieve this purpose.

Graphene oxide (GO) is the product of oxidation of graphite by strong oxidizing agents. The process introduces oxygenated functionalities that expand the layer separation; make the layers more irregular, and make the material an insulator as a result of distortion of the sp² network [6]. Reduction of graphene oxide to reduced graphene oxide (rGO) can recover the electrical conductivity and honeycomb structure. Reduced graphene oxide has a higher carrier mobility and optical transparency compared to the oxidized form [7]. Kamat et al. showed that for reduction of GO to rGO by TiO₂, TiO₂ acts as a UV sensitive photocatalyst due to having a large band gap. In this case, the highly negative conduction band edge energy of TiO₂ in comparision to that of carbon results in electron transfer from TiO_2 to carbon [8,9]. In the presence of ethanol, these electrons interact with the extended epoxy and carboxyl groups of GO and reduce it to rGO, while the holes are scavenged to produce ethoxy radicals [9]. We show that this gradual change in the conductivity in response to photoreduction of GO to rGO can be applied as a new strategy for real time monitoring of UV exposure time.





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Herein, we report a study of changes in the resistance of GO sheets decorated with TiO_2 nanoparticles during exposure to the UV radiation. To do this, these materials were incorporated in the electrical circuit by AC electrophoretic deposition on interdigitated electrodes. A feature of this deposition method is that it enables self-alignment of GO sheets and TiO_2 nanoparticles on the interdigitated electrodes.

2. Experimental methods

2.1. Materials

All chemicals were of analytical grade and used without further purification. TiO₂ (Aeroxide[®] P25, anatase/rutile, 21 nm particle size (TEM), \geq 99.5% trace metals basis), synthetic graphite powder (<20 µm, synthetic), sodium nitrate and potassium permanganate were purchased from Sigma-Aldrich (Sydney-Australia). 30% Hydrogen peroxide was supplied by Thermo Fisher Scientific Australia. Hydrochloric acid, acetone and ethanol were obtained from Chem-Supply Pty Ltd Australia. In all experiments where applicable, Milli-Q grade reagent water (18 M Ω cm) was used.

2.2. Sensor fabrication

GO sheets were synthesised by modified Hummer method [10] (see Section 1.1 and 1.2 in Supplementary Information for details). For sensor fabrication, 0.4 mg of TiO₂ nanopowder and 0.3 mg of GO sheets were suspended in 10 mL ethanol through 15 min ultrasonication. The deposition was performed using AC electrophoresis on gold-on-glass interdigitated concentric electrodes with 10 μ m electrode gap (DropSens, 33428 Llanera, Spain). The deposition set-up consisted of a waveform generator (Siglent SDG805 5 MHz from TRIO test & measurement) and the interdigitated electrodes. The condition for AC electrophoretic deposition was voltage of 20 V and frequency of 10 kHz. Electronic characterization was performed using a CHI660D potentiostat (CH Instruments, Texas, USA, operating with the CHI Software version 11.13).

2.3. UV and visible exposure experiments

An OSRAM ultra-vitalux 300 W 230 V E27 lamp with an intensity of $3200 \,\mu$ W/cm² was used for UV exposure experiments. It emits wavelengths in the range of $300-1000 \,\text{nm}$ to simulate solar radiation on a sunny day. The intensity of UV lamp was monitored using UV digital light meter (General Tools & Instruments UV513 AB Digital UVC Meter, 280–400 nm). The 150 W halogen lamp with wavelengths higher than 400 nm was used as a visible light source.

3. Results and discussion

Fig. 1 presents a schematic illustrating how we incorporate the sensing materials GO and TiO_2 into the interdigitated electrodes and study the change in the resistance of the GO sheets decorated with TiO_2 nanoparticles during UV exposure. The AC electrophoretic deposition is performed with sinusoidal voltage of 20 V at 10 kHz (Fig. 1a). Changes in the resistance of the GO sheets decorated with TiO_2 nanoparticles during UV exposure were measured using a potentiostat (Fig. 1b). The change in resistance is attributed to the transfer of photogenerated electrons from TiO_2 to epoxy and carboxyl moieties on the GO with the concomitant reduction of GO to rGO [8] (Fig. 1c).

The reduction of GO to rGO by TiO_2 was confirmed by the Raman spectra, as shown in Figure S. 1. The addition of TiO_2 to GO results in distortion of the GO structure (see TEM in Figure S. 2a-c), with exposure to UV radiation restoring the hexagonal structure of GO. This is not the case for the UV exposure of the GO itself (see Raman spectra in Figure S. 3).

Fig. 2a shows the SEM image of our sensor fabricated by AC electrophoretic deposition from ethanol. In this image, the region between the electrodes is filled successfully by GO sheets and TiO_2 nanoparticles. The alignment of GO sheets and TiO_2 nanoparticles between gold electrodes and along the field lines is due to dielectrophoresis [11–14]. At frequencies as high as 10 kHz, the electric field is the driving force for the deposition, as there is not enough time for particles and ions to migrate in each cycle. Therefore, electric field pushes charged particles toward the area where the density of the electric field lines is higher (see SEM image with higher magnification and microscopy image in Figure S. 4a and b, respectively).

The electrical measurements of this electrode are shown in Fig. 2b. The I-V curve in Fig. 2b shows an ohmic behavior. There is a major change in the slope of the *I-V* curve after UV exposure due to photoreduction of GO to rGO giving a change in resistance from 192 k Ω to 0.84 k Ω . Kamat et al. [8] reported a decrease in the resistance of GO-TiO_2 films on gold substrates from 233 $k\Omega$ to 30.5 k Ω after irradiation to UV for 2 h. They ascribed this change to the reconstruction of the sp² network in rGO. They related the lower resistance of unreduced GO-TiO₂, 233 Ω , in comparison to the other reports with lateral resistance of 32 M Ω for unreduced GO to the contribution of TiO₂ as well as having thicker film thicknesses. The chemical reduction of the GO film with the resistance of 32 M Ω resulted in the rGO film with $12 \text{ k}\Omega$ resistance across a 2 mm gap with 3 mm thickness [15]. Here, with the longer exposure times to UV radiation - around 36 h, which resulted in more conversion of GO to rGO and consequently lower resistance than the ones reported for photoreduction and chemical reduction of GO was

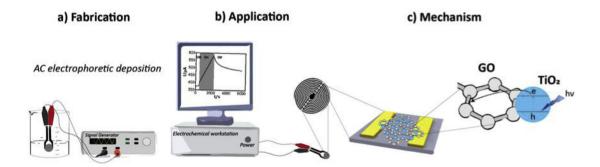


Fig. 1. Scheme of the a) fabrication, b) application and c) mechanism of the UV sensor based on GO/TiO₂. The interdigitated electrode is immersed in a dispersion of TiO₂/GO in either ethanol or acetone and connected to the AC signal generator, which applies sinusoidal voltage of 20 V at 10 kHz. The sensor behavior under UV radiation is studied by *I*-*t* and *I*-*V* curves. The conductance change of the sensor is attributed to the transfer of photogenerated electrons from the TiO₂ to epoxy and carboxylate moieties on the GO with the concomitant reduction of GO to rGO. (A colour version of this figure can be viewed online.)

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