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Morphological, Structural and Optical Studies of Graphene Oxide Thick Films Fabricated by Screen Printing

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ABSTRACT

Graphene oxide (GO) thick films were successfully fabricated on glass substrates using a simple and cost-effective screen-printing technique and systematically investigated for their morphological, structural and optical properties. Scanning electron microscopy revealed a crumpled, wrinkled, and flake-like morphology with loosely stacked GO sheets, forming a porous surface network. The specific surface area measured by the Brunauer–Emmett–Teller (BET) method was found to be 3.29 m^2/g, indicating partial restacking of GO layers in the thick-film configuration. Energy-dispersive X-ray spectroscopy confirmed the presence of carbon and oxygen elements, verifying the successful formation of graphene oxide without detectable impurities. X-ray diffraction analysis showed a characteristic (001) diffraction peak of GO, consistent with JCPDS data, confirming the layered structure with expanded interlayer spacing. The average crystallite size, calculated using the Debye–Scherrer equation, was found to be 65.84 nm.Fourier transform infrared spectroscopy revealed prominent oxygen-containing functional groups such as hydroxyl, epoxy, carbonyl, and alkoxy groups, confirming the oxidized nature of the GO films. UV–Visible absorption studies exhibited a strong absorption peak in the ultraviolet region attributed to π–π* transitions, along with a shoulder corresponding to n–π* transitions of C=O bonds. The optical band gap ~3.2–3.6 eV estimated using the Tauc method indicated the semiconducting nature of the GO thick films. The combined results demonstrate that screen-printed graphene oxide thick films possess suitable structural and optical characteristics for potential applications in optoelectronics, sensing, and energy-related devices.

1. Introduction

Graphene oxide (GO), a chemically modified derivative of graphene, has attracted significant attention due to its unique structural, optical, and physicochemical properties. Derived from graphite through oxidative exfoliation, GO consists of a single or few layers of carbon atoms arranged in a hexagonal lattice, decorated with abundant oxygen-containing functional groups such as hydroxyl, epoxy, carbonyl, and carboxyl moieties. These functional groups disrupt the extended π-conjugation of graphene, imparting hydrophilicity, tunable band structure, and enhanced chemical reactivity. As a result, GO has emerged as a versatile material for applications in sensors, optoelectronic devices, energy storage systems, membranes, and environmental remediation [1].

The performance of GO-based devices is strongly influenced by the morphology, thickness, crystallinity, and optical characteristics of the deposited films. Thick films, in particular, are advantageous for large-area devices, robust sensing platforms, and applications requiring higher material loading. Compared to thin films, thick films can provide improved mechanical stability, enhanced light absorption, and greater interaction volume with external stimuli such as gases, light, or liquids. Therefore, understanding the morphological, structural, and optical properties of GO thick films is essential for optimizing their functionality in practical applications. Among various fabrication techniques, screen printing stands out as a simple, cost-effective, and scalable method for depositing thick films on diverse substrates. Screen printing enables precise control over film thickness, uniformity, and pattern geometry, making it highly suitable for industrial-scale production. The technique also allows the use of GO inks or pastes prepared in aqueous or low-toxicity solvents, aligning well with green and sustainable manufacturing principles. Moreover, screen printing is compatible with flexible and rigid substrates, broadening the scope of GO-based device integration. Morphological analysis of screen-printed GO thick films provides insight into surface roughness, flake distribution, porosity, and film continuity, which directly affect charge transport, optical absorption, and interfacial interactions. Structural studies, typically carried out using X-ray diffraction and spectroscopic techniques, help elucidate the degree of oxidation, interlayer spacing, crystallite size, and disorder within the GO framework [2]. These structural parameters are closely linked to the electronic structure and defect density of the material. Optical studies, including UV–visible spectroscopy, are crucial for understanding light–matter interactions in GO films, such as absorption behavior, optical band gap, and electronic transitions associated with oxygen functional groups and localized sp^2 domains.

In this context, the present work focuses on the fabrication of graphene oxide thick films using the screen-printing technique and a comprehensive investigation of their morphological, structural, and optical properties. By correlating the fabrication process with the resulting material characteristics, this study aims to provide valuable insights into the suitability of screen-printed GO thick films for optoelectronic, sensing, and environmental applications. The findings are expected to contribute to the development of low-cost, scalable, and high-performance GO-based devices [3].

2. Experimental Methods

2.1 Materials

Natural graphite powder (purity ≥99%) was used as the precursor for the synthesis of graphene oxide (GO). Analytical-grade sulfuric acid (H_2SO_4), phosphoric acid (H_3PO_4), potassium permanganate ($KMnO_4$), hydrogen peroxide (H_2O_2), and hydrochloric acid (HCl) were employed for the oxidation and purification processes. Deionized (DI) water was used throughout the experiments. All chemicals were used as received without further purification. Glass substrates were selected for thick film

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deposition due to their good thermal stability and smooth surface. Organic binders and solvents suitable for screen-printing paste preparation were used to ensure proper viscosity and adhesion [4].

2.2 Synthesis of Graphene Oxide

Graphene oxide was synthesized from natural graphite powder using a modified Hummers' method. In a typical synthesis, graphite powder was added slowly to a mixture of concentrated H_2SO_4 and H_3PO_4 under continuous stirring in an ice bath to maintain low temperature. $KMnO_4$ was gradually introduced as an oxidizing agent, ensuring controlled oxidation and preventing overheating. The reaction mixture was stirred for several hours until a thick brown slurry was obtained, indicating effective oxidation of graphite layers [5].

After completion of the reaction, the mixture was diluted with DI water followed by the slow addition of H_2O_2 to terminate the oxidation process. The resulting suspension was repeatedly washed with dilute HCl and DI water through centrifugation until a neutral pH was achieved. The obtained GO was then dried at low temperature and subsequently dispersed in DI water using ultrasonication to form a stable GO suspension.

2.3 Preparation of GO Screen-Printing Paste

To fabricate thick films, a printable GO paste was prepared by mixing the aqueous GO dispersion with a suitable organic binder and solvent system. The mixture was magnetically stirred and ultrasonicated to achieve uniform dispersion and appropriate rheological properties required for screen printing. The viscosity of the paste was optimized to ensure smooth transfer through the screen mesh and uniform film formation on the substrate [6].

2.4 Fabrication of GO Thick Films by Screen Printing

GO thick films were deposited on pre-cleaned glass substrates using a manual screen-printing setup. Prior to printing, the substrates were ultrasonically cleaned in acetone, ethanol, and DI water, followed by drying in air. A stainless-steel or nylon mesh screen with a defined mesh size was used to control film thickness. The prepared GO paste was placed on the screen, and a rubber squeegee was used to spread the paste uniformly across the substrate. Multiple printing passes were carried out to achieve the desired film thickness. After printing, the films were dried at room temperature to remove excess solvent and then annealed at moderate temperature to improve film adhesion and remove residual organic components [7].

2.5 Characterization Techniques

The surface morphology and microstructural features of the screen-printed GO thick films were examined using scanning electron microscopy (SEM). The analysis provided information on flake distribution, surface roughness, porosity, and film uniformity [8]. Structural characterization was performed using X-ray diffraction (XRD) with Cu Kα radiation (λ = 1.5406 Å). The diffraction patterns were analyzed to determine interlayer spacing, crystallinity, and the degree of oxidation of GO. Functional groups and bonding configurations were further confirmed using Fourier transform infrared (FT-IR) spectroscopy [9]. Optical properties of the GO thick films were investigated using UV–visible spectroscopy in the wavelength range of 200–800 nm. The absorption spectra were used to study electronic transitions and estimate the optical band gap using Tauc plots. This systematic materials and methods approach ensures reproducible fabrication and reliable evaluation of the morphological, structural, and optical properties of screen-printed graphene oxide thick films [10].

3. Results and Discussion

3.1 Structural Analysis

The structural properties of the GO thick films were investigated using X-ray diffraction (XRD), shown in Fig. 1. The XRD pattern shows a prominent diffraction peak around 2θ ≈ 10–11°, which corresponds to the (001) plane of graphene oxide. This peak confirms the successful oxidation of graphite and the formation of GO. The increased interlayer spacing compared to pristine graphite is attributed to the intercalation of oxygen functional groups and water molecules between the graphene layers. The broad nature of the diffraction peak indicates reduced crystallinity and the presence of structural disorder, which is typical for GO materials. The absence of significant reduction of the graphite (002) peak around 26° further confirms the effective exfoliation and oxidation of graphite into GO sheets. These structural features play a crucial role in determining the electronic and optical behavior of the thick films. The average crystallite size, calculated using the Debye–Scherrer equation, was found to be 65.84 nm, indicating the formation of well-defined crystalline domains within the GO layers. This nanoscale crystallite size suggests an ordered layered structure while maintaining sufficient structural defects associated with oxygen functional groups.

Fourier transform infrared (FT-IR) spectroscopy further supports the formation of GO by revealing characteristic absorption bands corresponding to oxygen-containing functional groups. The broad absorption band around 3200–3400 cm^{-1} is attributed to O–H stretching vibrations, while peaks near 1720 cm^{-1} and 1620 cm^{-1} correspond to C=O stretching and C=C skeletal vibrations, respectively. Additional peaks in the range of 1000–1300 cm^{-1} are associated with C–O stretching vibrations of epoxy and alkoxy groups. These results confirm the successful functionalization of graphene sheets during oxidation.

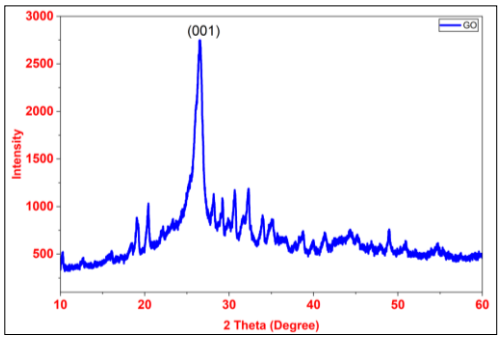

Fig. 1 XRD spectrum of screen printed GO film

3.2 Morphological Analysis

The surface morphology of the screen-printed graphene oxide (GO) thick films was examined using scanning electron microscopy (SEM) and shown in Fig. 2. The SEM micrographs reveal that the films are continuous and uniformly distributed over the substrate surface, confirming the effectiveness of the screen-printing technique for thick film fabrication. The GO sheets appear as randomly oriented, wrinkled, and overlapping flakes, which is a characteristic feature of exfoliated GO. Such wrinkling and folding arise from the presence of oxygen-containing functional groups and prevent the restacking of graphene layers.

The observed porous and rough surface morphology is beneficial for applications such as gas sensing and photocatalysis, as it provides a high surface-to-volume ratio and abundant active sites. The absence of major cracks or pinholes in the films indicates good adhesion between the GO layer and the glass substrate. Additionally, multiple printing passes result in increased film thickness and improved surface coverage, demonstrating that film morphology can be tuned by controlling the printing parameters.

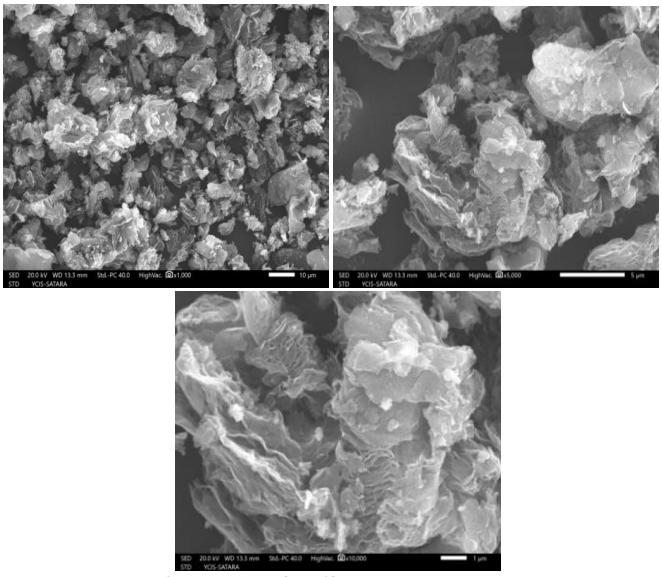

Fig. 2 SEM images of screen printed GO film

Energy-dispersive X-ray spectroscopy confirmed the presence of carbon and oxygen as the dominant elements, verifying the successful formation of graphene oxide. The absence of additional elemental peaks indicates high purity of the film and confirms that no residual impurities or foreign elements were introduced during synthesis.

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The specific surface area measured by the Brunauer–Emmett–Teller (BET) method was found to be 3.29 m^2/g (Fig. 3). BET surface area of graphene oxide (GO) typically ranges from 100 to over 1000 m^2/g depending on preparation methods. This relatively low surface area, compared to typically reported values for exfoliated graphene oxide powders, indicates partial restacking and aggregation of GO sheets in the thick-film configuration. During the screen-printing and subsequent drying process, strong interlayer Van der Waals interactions and hydrogen bonding between oxygen-containing functional groups promote the reassembly of individual GO layers into a more compact structure. As a result, a significant fraction of the internal surface area becomes inaccessible to nitrogen adsorption during BET analysis.

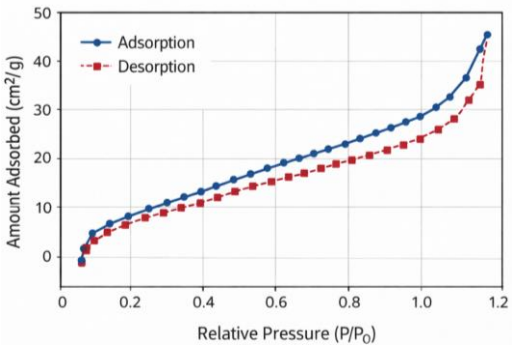

Fig. 3 BET surface area analysis of GO

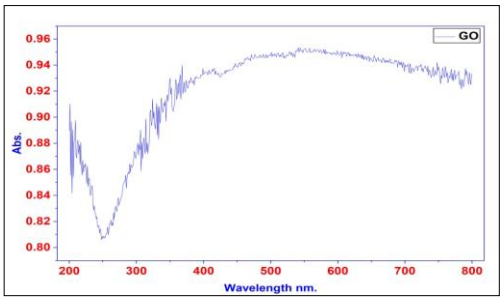

Fig. 4 UV-Vis spectrum of screen printed GO film

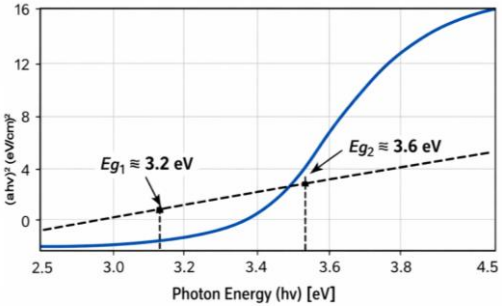

Fig. 5 Band gap graph of synthesised GO

3.3 Optical Analysis

The optical properties of the screen-printed GO thick films were studied using UV–visible spectroscopy (Fig. 4). The absorption spectra show a strong absorption peak in the ultraviolet region around 230–240 nm, which is attributed to π–π* transitions of aromatic C=C bonds in the sp^2 carbon network. A shoulder or weak absorption feature observed near 300 nm corresponds to n–π* transitions associated with C=O bonds of oxygen functional groups. The optical band gap value, estimated using the Tauc method, was found to be in the range of ~3.2–3.6 eV (Fig. 5), indicating the semiconducting nature of the GO thick films. The calculated band lies in the range typically reported for graphene oxide, indicating semiconducting behaviour. The presence of oxygen functional groups and disrupted sp^2 domains leads to the opening of a band gap, which is absent in pristine graphene. This tunable optical band gap makes GO thick films suitable for optoelectronic and sensing applications.

3.4 Correlation between Morphology, Structure, and Optical Properties

The combined morphological, structural, and optical analyses demonstrate a strong interdependence between film fabrication and material properties. The wrinkled and porous morphology enhances light absorption and increases defect density, while the expanded interlayer spacing and functional groups confirmed by XRD and FT-IR significantly influence the electronic structure of GO. These factors collectively contribute to the observed optical behaviour and semiconducting nature of the films. Overall, the results confirm that screen printing is an effective and scalable technique for fabricating uniform GO thick films with desirable morphological, structural, and optical characteristics. Such films hold significant potential for use in low-cost optoelectronic devices, sensors, and environmental remediation technologies.

4. Conclusion

In the present study, graphene oxide (GO) thick films were successfully fabricated on glass substrates using a simple, cost-effective, and scalable screen-printing technique. The results clearly demonstrate that screen printing is a suitable method for producing uniform and mechanically stable GO thick films, which are essential for large-area and device-oriented applications.

Morphological investigations revealed that the screen-printed GO films possess a continuous, wrinkled, and porous surface composed of randomly oriented and overlapping GO sheets. This morphology provides a high surface area and abundant active sites, which are highly advantageous for applications such as gas sensing, optoelectronics, and environmental remediation. The absence of significant cracks or discontinuities confirms good film adhesion and uniformity. Structural analysis using X-ray diffraction confirmed the successful oxidation and exfoliation of graphite into graphene oxide, as evidenced by the characteristic (001) diffraction peak and increased interlayer spacing.

FT-IR spectroscopy further verified the presence of oxygen-containing functional groups, confirming effective functionalization of the graphene sheets. These structural features play a crucial role in tailoring the electronic and optical behavior of GO thick films. Optical studies showed strong ultraviolet absorption arising from π–π* and n–π* electronic transitions, indicating disrupted sp^2 domains and semiconducting behavior. The estimated optical band gap ~3.2–3.6 Ev highlights the potential of GO thick films for optoelectronic and sensor-based applications, where tunable optical properties are essential.

Overall, the strong correlation between morphology, structure, and optical properties confirms that the screen-printed GO thick films exhibit characteristics favorable for practical device fabrication. This work provides valuable insights into the fabrication and characterization of graphene oxide thick films and establishes a foundation for their future use in low-cost, scalable, and high-performance electronic, sensing, and environmental applications.

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