Micro-Raman Spectroscopy for Graphene Characterization

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There has been growing interest in combining the unique properties of graphene-based materials with silver nanostructures, aiming for their utilization in the fields of plasmonics and metamaterials applications. Silver nanoparticles and nanostructures, with localized surface plasmonic resonance spanning from the visible to the near-infrared range, find utility in various applications, particularly in surface-enhanced Raman scattering (SERS). Materials based on graphene, for instance, graphene oxide (GO) and reduced graphene oxide (RGO), have demonstrated their potential as SERS substrates by generating strong chemical enhancement.

graphene graphene oxide micro-Raman

1. Introduction

Graphene exhibits special physical and electrical properties. It demonstrates broad light absorption, a linear dispersion band structure, significant charge-carrier mobility, and remarkable optical qualities. For instance, it offers high transparency within the visible spectrum [1], photo-response capabilities extending into the Terahertz frequency range, and customizable infrared optical absorbance [2]. Furthermore, its unique band structure and electron transport properties make it a promising candidate for developing high-performance modulators, sources, and detectors designed for operation in the infrared (IR) segment of the electromagnetic spectrum [3]. The optical characteristics of graphene films vary significantly depending on the fabrication method used, whether it is exfoliation or chemical vapor deposition (CVD) [4]. CVD, a method intended for producing high-quality few-layer graphene, has been successfully applied in ultradense photonic, optoelectronic, and electronic devices, as described in previous research [5][6].

2. Structure of Graphene, Graphene Oxide, and Reduced Graphene Oxide

Graphene consists of a single layer of carbon atoms arranged in a hexagonal lattice and originates from graphite [7]. Its unique properties include sp^2 hybridization and an incredibly slim atomic thickness measuring 0.345 nm [8]. A perfect graphene sheet has a flat structure with minor ripples, and its carbon atoms form a trigonal pattern [9]. Various methods have been developed to fabricate graphene, including CVD [10], epitaxial growth [11], micromechanical exfoliation [12], and epitaxial growth on insulating surfaces like SiC [13]. These techniques have significantly advanced research on monolayer graphene.

GO, on the other hand, is a derivative of graphene that contains oxygen-functional groups like epoxide, carbonyl, carboxyl, and hydroxy [14]. These groups render GO sheets water-soluble; their removal leads to flocculation and precipitation. GO features a complex structure with oxygen-rich domains interspersed among areas where carbon atoms retain their sp^2 hybridization [15].

Graphite and graphite oxide, although related, have distinct properties [16]. Graphite oxide is created by oxidizing graphite, which introduces oxygen functionalities that increase the distance between layers and make the material water-dispersible [17]. Exfoliation of graphite oxide through sonication in water results in the formation of GO [18]. The primary difference between graphite oxide and GO is in the number of layers: graphite oxide is multilayered, while GO dispersions mainly consist of monolayer flakes, with some multilayer flakes present [19].

Zeta potential measurements show that GO sheets in water carry negative charges [20], which leads to electrostatic repulsion among the sheets and results in a stable aqueous suspension. The utilization of organic compounds in chemically altering GO sheets can lead to the attainment of uniform dispersions in organic solvents [21].

A widely employed technique for synthesizing GO is the adapted Hummers method, which relies on oxidizing graphite by using a blend of concentrated sulfuric acid, sodium nitrate, and potassium permanganate [22]. While the exact chemical arrangement of GO remains not entirely comprehended, it is considered partially amorphous, with proposed structural models consisting of hydroxyl and epoxy functional groups [23].

Various techniques, such as drop-casting [24], dip-coating [25], spraying [26], spin-coating [27], and electrophoresis [28], can be used to deposit GO sheets as thin films on different substrates.

GO and graphene have also been extensively used to obtain composite coatings, enhancing the wear behavior and other mechanical properties of the substrate. For example, in Ref. [29], an enhanced wear behavior in nickel/graphene composite coatings is reported. Similarly, in Ref. [30], the properties of nickel-based composite coatings modified with GO are investigated.

The electrical properties of GO sheets differ significantly from those of pristine graphene. As a consequence, films composed of GO tend to manifest resistance values that commonly exceed 10^{12} Ω/sq [31].

A partial reduction of GO can yield sheets resembling graphene by eliminating oxygen-related groups [32]. Chemical reduction methods typically use reducing agents such as hydrazine or involve creating highly alkaline conditions [33]. Ascorbic acid has emerged as a promising alternative to hydrazine [34]. The thermal reduction process for GO is commonly conducted at temperatures exceeding 200 °C within inert or reducing surroundings, with an enhanced efficacy observed at elevated temperatures [35].

These reduced GO sheets exhibit reduced hydrophilicity and display a propensity to aggregate within the solution, stemming from the absence of oxygenated functionalities [36]. The chemical reduction of GO partially restores its conductivity [37], yet it remains significantly inferior by several orders of magnitude when compared to the pristine

graphene counterpart. The degree of this reduction determines the conductivity of the resulting RGO, which can range from 0.05 to 500 S/cm and is intricately linked to the proportion of graphitic (sp^2) to oxidized (sp^3) zones [38].

3. State of the Art

Raman spectroscopy has become an indispensable tool for comprehensively characterizing graphene and its derivatives like GO and RGO [39][40]. The technique's ability to identify characteristic vibrational modes in materials offers an understanding of the structural and chemical compositions of these graphene-based materials [41]. For example, Neeraj Sharma and colleagues [42] showed that the presence or absence of functional groups significantly alters the material's vibrational modes, evident from changes in the Raman spectra [41]. This is particularly valuable for researchers aiming to tailor the properties of graphene-based materials for specific applications, such as optoelectronics or sensing.

The G and D peaks in the Raman spectra serve as critical markers for the structural integrity and chemical modifications of graphene-based materials [39]. The G peak, a fundamental graphitic peak, is ubiquitous in all hexagonal graphitic structures, while the D peak arises due to imperfections and impurities introduced by functional groups [39].

Jiang-Bin Wu and colleagues [43] further elaborate on how Raman spectroscopy can study the impact of various external factors like doping agents, mechanical forces, and environmental conditions on single-layer graphene. Additionally, the authors provide insights into the identification of material defects and layer counts through Raman spectral features.

K. Kanishka H. De Silva et al. [44] further extend this discussion by emphasizing the challenges posed by the abundant lattice defects in GO and RGO. Their work provides new insights into the optical visibility and Raman signals of these materials when deposited on different Si-based substrates. They found that dielectric substrates with lower reflectance in the visible range offer better optical contrast and more intense Raman signals. This advancement is significant because it implies that substrate choice can affect the quality of Raman spectroscopic data and, consequently, our understanding of the material's structural and optical properties. Moreover, De Silva et al. [44] introduced the importance of the D' band in the Raman spectra for a more comprehensive understanding of the structural variations in monolayer flakes of GO and RGO.

4. Micro-Raman Spectroscopy for Graphene Characterization

Raman spectroscopy is a critical tool for characterizing the properties of graphene, especially in the development of graphene-based field-effect transistors (FETs). By identifying the doping levels, Raman spectroscopy enables the fine-tuning of the electronic properties of these transistors, directly affecting their speed and energy efficiency. Additionally, the technique provides valuable data on heat generation and dissipation within the FETs, contributing to their long-term stability and performance [45][46][47][48].

The technique also plays a significant role in advancing energy storage devices. Specifically, it helps researchers understand the distortion of electrode material during charge and discharge processes. This understanding is vital for enhancing both the efficiency and longevity of batteries and other energy storage systems based on graphene [49].

In optoelectronic applications like solar cells and organic light-emitting diodes (OLEDs) [50], graphene's high optical transparency, electrical conductivity, and mechanical flexibility make it a material of choice. Raman spectroscopy aids in the identification of interlayer coupling and the number of graphene layers in these devices, which is crucial for optimizing their performance.

Moreover, in nanoelectromechanical systems (NEMS), the technique is used to probe the local stress within multilayer graphene cantilevers [51].

Raman spectroscopy is also instrumental in the study of van der Waals 2D heterostructure devices, such as those combining graphene and MoS_2 [52]. The method allows for the in situ examination of charge transfer within these heterostructures, providing essential insights into the electronic interplay between the various layers.

While the applications of Raman spectroscopy in areas like FETs, energy storage, and optoelectronics are well-established, its potential in sensing technologies is gaining increasing attention [53].

It is worth noting that MoS_2, a prominent member of the transition metal dichalcogenides family, has demonstrated significant applicability across various technological domains. Notably, MoS_2 has been studied for its role as a spin-valley filter, as outlined in [54]. Additionally, its intrinsic optoelectronic characteristics make it suitable for phototransistor devices, as detailed in [55]. MoS_2 has also been explored for its potential in rectifying contacts, as reported in [56], and in tunneling field-effect transistors, as described in [57].

The study of transition metal dichalcogenide monolayers (MX_2), including MoS_2, has revealed the importance of metal-induced gap states (MIGS) in spin-valley filters [54]. These MIGS are predicted to mediate valley- and spin-resolved charge transport near the ideal electrode/MX_2 interface, thus initiating filtering. This insight is particularly crucial when the channel length is diminished as MIGS begin to govern the overall valley-spin transport in the tunneling regime. The findings offer design guidelines for efficient valley-spin filter devices, which could revolutionize information processing technologies.

In addition to its role in valley-spin filtering, MoS_2 has been extensively studied for its intrinsic optoelectronic characteristics [55]. Raman spectroscopy serves as a vital tool for understanding these properties, especially when MoS_2 is integrated into functional devices like phototransistors. Utilizing a fully transparent van der Waals heterostructure, researchers have been able to reveal the intrinsic photoresponsivity characteristics of monolayer MoS_2, including its internal responsivity and quantum efficiency.

Similarly, transition metal dichalcogenides (TMDs), particularly MoS_2, have garnered attention for their semiconducting applications at the nanoscale. These materials exhibit direct band gaps and high charge mobilities,

making them ideal for use in nanoscale devices. One of the critical aspects of TMDs is the understanding of Schottky barrier heights (SBHs) [56], which are crucial for efficient charge injection into these semiconductors. These theoretical models help in estimating the charge neutrality levels and canonical Schottky barrier heights, which are pivotal for the device's performance.

Further exploring the potential of MoS_2, tunneling field-effect transistors (TFETs) using monolayer MoS_2 as the channel have emerged as promising contenders for low-energy electronics. These MoS_2-TFETs [57] are compatible with silicon CMOS technology and exhibit a lower off-state leakage current and subthreshold swing compared to traditional MOSFETs.

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