JOINT INSTITUTE FOR NUCLEAR RESEARCH

Frank Laboratory of Neutron Physics, Sector of Raman Spectroscopy

FINAL REPORT
ON START Program

Raman Spectroscopic Characterization of Graphene Oxide

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Contents

Abstract

This report presents the Raman spectroscopic analysis of two graphene oxide (GO) samples conducted during a practical internship at the Joint Institute for Nuclear Research (JINR). Raman spectroscopy is a non-destructive technique used to probe the structural integrity and defect density of carbon-based materials. GO, a chemically modified form of graphene, contains oxygen functionalities that introduce structural disorder, which can be assessed using Raman features such as the D and G bands.

Two samples were investigated (Sample 1 and Sample 2) under a 633 nm excitation source using a Confotec CARS Raman spectrometer. Sample 1 exhibited a broader D band with a full width at half maximum (FWHM) of 49.55 cm^-1 compared to 37.92 cm^-1 for Sample 2, indicating higher structural disorder. Despite this, the I_D/I_G ratios were 0.47 and 0.59 for Sample 1 and 2, respectively. This suggests a non-linear correlation between D-band intensity and disorder, possibly due to differences in oxidation levels or structural amorphization. Unique Raman features, such as a 643 cm^-1 band exclusive to Sample 1were also observed.

The findings demonstrate the value of Raman spectroscopy for evaluating graphene oxide microstructure and provide insight into the complexity of defect characterization in oxidized carbon materials.

1. Introduction

1.1 Raman spectroscopy

Raman spectroscopy is a powerful analytical technique that exploits the scattering of monochromatic light to provide information about the vibrational and rotational transitions in materials. The phenomenon was first observed by Indian physicist C.V. Raman and independently by Soviet scientists L.I. Mandelstam and G.S. Landsberg, leading to the identification of Raman scattering.

The core principle of Raman spectroscopy involves inelastic scattering of light, where incident photos interact with molecular vibrations, resulting in a shift in energy. This energy change manifests as distinct Raman shifts in the spectrum, which can be used to determine the chemical structure, composition, and homogeneity of various materials—be they solid, liquid, or gas.

Key components of a Raman spectrometer include a laser source for sample excitation, an injection/rejection filter to manage light pathways, a spectrograph and detector for wavelength dispersion and intensity measurement, a microscope for focusing laser light on the sample surface, and computer systems for data control and analysis.

By analyzing Raman spectra—characterized by specific bands whose shifts and intensities reflect material properties, researchers can gain insight into stress variations and crystallinity within samples, making Raman spectroscopy an invaluable tool across numerous scientific fields [1].

Figure 1. *General view on the multifunctional 3D Scanning Laser Microscope – "Confotec CARS".*

1.2 Overview of Graphene Oxide (GO)

Graphene oxide (cross) is a spinoff of graphene, composed of a single layer of carbon atoms arranged in a -dimensional honeycomb lattice, functionalized with various oxygen-containing agencies which include hydroxyl, epoxy, and carboxyl businesses. Those functional companies adjust the electronic, mechanical, and chemical residences of graphene, making pass tremendously dispersible in water and well suited with an extensive range of composite materials [2]. Due to its tunable surface chemistry cross is widely utilized in programs along with sensors, power storage gadgets, flexible electronics, and biomedical engineering [3].

1.3 Importance of Raman Spectroscopy in Carbon-Based Materials

Raman spectroscopy is a popular and efficient method for describing materials based on carbon, particularly graphene and its derivatives. It is a non-destructive optical method that offers comprehensive details about the atoms' vibrational modes within a substance [4]. Raman spectroscopy can be used to analyze the number of layers, structural disorder, defect density, and chemical changes in the case of GO. The G band (~1580 cm^{-1}), which corresponds to the in-plane vibration of sp^2-hybridized carbon atoms, and the D band (~1350 cm^{-1}), which is linked to defects and disorder, are important characteristics in the Raman spectrum of GO. The degree of disorder inside the graphene structure is often measured using the intensity ratio of the D and G bands (ID/IG) [5].

1.4 Objective and Scope of the Practice at JINR

This report presents the work conducted during a practical internship at the Raman Sector of the Joint Institute for Nuclear Research (JINR), Dubna, Russia. The primary objective of the practice was to perform Raman spectroscopic analysis of graphene oxide samples to investigate their structural and defect-related properties. The experimental work involved using a Raman spectrometer to record spectra from GO samples under controlled conditions and analyzing the resulting data to extract meaningful physical parameters. The practice aimed to provide hands-on experience with Raman instrumentation, sample handling, data processing, and interpretation of spectral features relevant to graphene oxide.

1.5 Significance of Study

Characterizing graphene oxide at the microstructural level is crucial for optimizing its performance in various scientific and technological applications. Raman spectroscopy offers a rapid and accurate means of evaluating GO quality, including the degree of oxidation and structural defects, which can directly influence its conductivity, mechanical strength, and chemical reactivity. By conducting Raman analysis at JINR, this study contributes to ongoing research in nanomaterials and strengthens the practical understanding of advanced characterization techniques. Moreover, the hands-on experience gained through this practice provides a strong foundation for further research or industrial applications involving carbon-based nanomaterials.

2 Materials and Methods

2.1 Raman Instrumentation

All Raman measurements were carried out at the **Raman Sector of the Joint Institute for Nuclear Research (JINR)** using an *Confotec CARS* Raman spectrometer. The system was equipped with a insert laser wavelength *633nm* laser source, which was focused on the sample through a magnification,*40x objective lens*.

The spectrometer was calibrated using a silicon standard, with the Si Raman peak at 520.7 cm^{-1} serving as a reference point. The Spectra were recorded in the range of *500–3000* cm^{-1}capturing the characteristic D, G, and 2D bands of graphene oxide.

2.2 Measurement Procedure

Before measurement, the sample surface visually inspected under the microscope to identify suitable regions with well-dispersed GO flakes. Raman spectra collected from multiple points across the sample to ensure reproducibility and account for any inhomogeneities. Each scan acquired with an integration time of 5,*10,20 and 30 seconds*.

The focus carefully adjusted to maximize the Raman signal from the GO layer. Data was saved for post-processing and quantitative analysis.

2.3 Data Processing

The collected spectra were processed using Origin *Lab*. Baseline correction was performed to remove fluorescence background, and peak fitting was applied using Lorentzian functions to determine the position, full width at half maximum (FWHM), and intensity of the D, G, and 2D bands.

The intensity ratio of the D and G peaks (ID/IG) was calculated to estimate the degree of disorder and defect density within the GO structure. Any shifts in peak positions were analyzed to detect potential strain or doping effects.

3 Results

3.1 Raman Spectra of GO

Raman spectroscopy was employed to characterize the structural properties of two graphene oxide (GO) samples, referred to as Sample 1 and Sample 2. The measurements were conducted using a 633 nm excitation laser, with a 40x/0.6 objective, a 60 0 grating, and an accumulation time of 50 seconds. A pinhole size of 100 was used to enhance spatial resolution.

The Raman spectra for both samples show characteristic bands associated with disordered carbon materials. Specifically, the D band (disorder-induced) and G band (graphitic) were observed in both spectra, providing key insight into the defect density and degree of graphitization of the samples. Notably, a D′ shoulder band and a D + D″ combination band were more prominent in Sample 1, indicating a higher concentration of structural defects.

3.2 Quantitative Analysis

To extract meaningful physical insights, a detailed analysis of the D and G bands was performed. The parameters of interest—peak position, full width at half maximum (FWHM), and intensity were measured using curve fitting techniques.

Table 1 summarizes typical spectral parameters for a selected measurement point:

Sample	**D band**	**G band**	**D’ band**	**2D band**	**?**	**D + D” band**
1	**1322**	**1567**	**1603**	**2639**	**643**	**2442**
2	**1320**	**1567**	**1603**	**2632**	**-**	**2443**

Table 2 FWHM Measurements of D and G Bands for Samples:

Sample	**D Band FWHM (cm⁻¹)**	**G Band FWHM (cm⁻¹)**
Sample 1	**49.55**	**131**
Sample 2	**37.92**	**110**

The broader FWHM values in Sample 1 suggest a higher level of disorder and defect density compared to Sample 2. Additionally, the presence of an unidentified band at 643 cm⁻¹—exclusive to Sample 1—may indicate defect-specific vibrational modes, though its exact origin remains unclear.

3.3 Figures

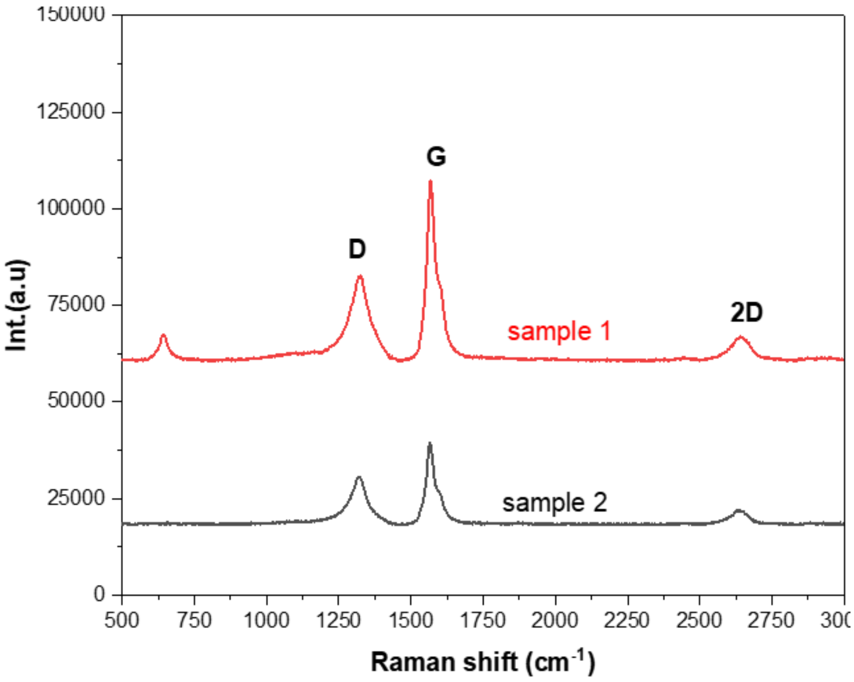


Figure 1: Raman spectrum of graphene oxide showing D, G, and 2D bands.

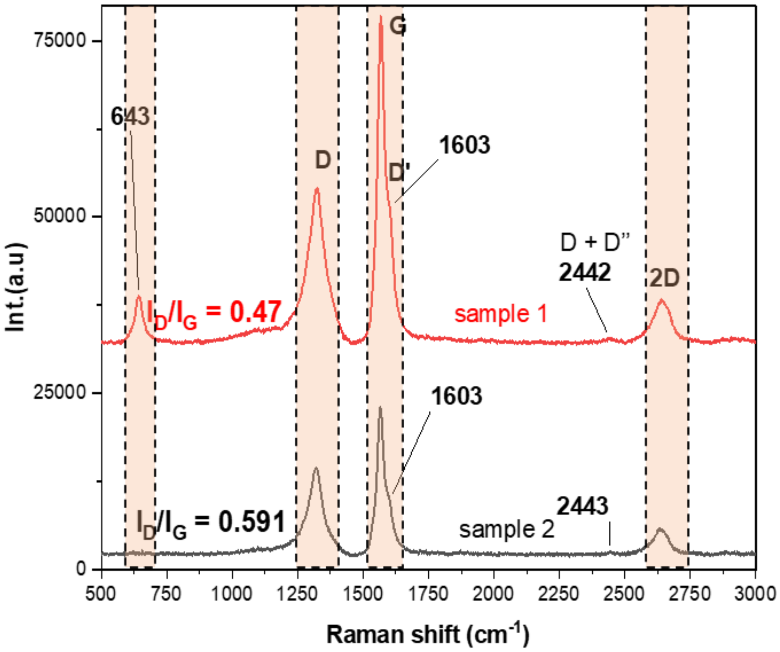
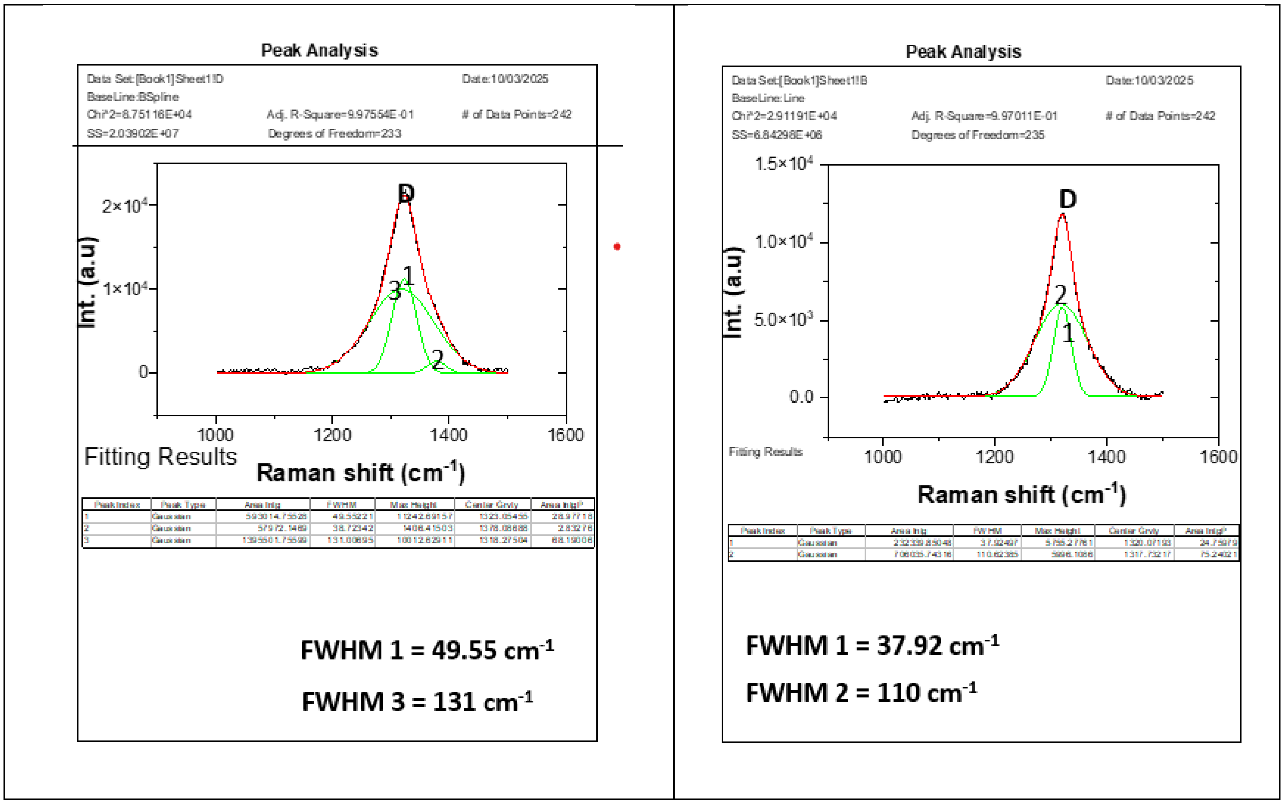


Figure 2: Raman spectra of GO Sample 2 with characteristic D and G bands**.**



Peak Analysis

Data Set:[Book1]Sheet1!D
Date:10/03/2025
BaseLine:BSpline
Chi^2=8.75116E+04
Adj. R-Square=9.97554E-01
# of Data Points=242
SS=2.03902E+07
Degrees of Freedom=233

Fitting Results

Peak Index	Peak Type	Area Intg	FWHM	Max Height	Center Grvty	Area IntgP
1	Gaussian	593014.75528	49.55221	11242.89157	1323.05455	28.97718
2	Gaussian	57972.1469	38.72342	1406.41503	1378.08888	2.83278
3	Gaussian	1395501.75599	131.00895	10012.82911	1318.27504	68.19006

FWHM 1 = 49.55 cm^{-1}

FWHM 3 = 131 cm^{-1}

Peak Analysis

Data Set:[Book1]Sheet1!B
Date:10/03/2025
BaseLine:Line
Chi^2=2.91191E+04
Adj. R-Square=9.97011E-01
# of Data Points=242
SS=6.84298E+06
Degrees of Freedom=235

Fitting Results

Peak Index	Peak Type	Area Intg	FWHM	Max Height	Center Grvty	Area IntgP
1	Gaussian	232339.85048	37.92497	5755.27781	1320.07193	24.75979
2	Gaussian	706035.74316	110.62385	5996.1086	1317.73217	75.24021

FWHM 1 = 37.92 cm^{-1}

FWHM 2 = 110 cm^{-1}

Figure 3: Comparative FWHM analysis of D and G bands for Samples 1 and 2 respectively.

4.Discussion

Raman spectroscopy is a key technique for characterizing structural defects and disorder in carbon-based materials like graphene oxide (GO). The primary features of interest in the spectra are the D band (∼1350 cm^{-1}), associated with breathing modes of sp^2 carbon rings and disorder, and the G band (∼1580 cm^{-1}), corresponding to the E2g phonon of sp^2 carbon atoms. The intensity ratio of these two bands (I_D/I_G) serves as a direct indicator of the degree of disorder and defect density in the material.

From the collected spectra, both GO1 and GO2 exhibit prominent D and G bands, confirming the presence of structural defects typical of oxidized graphene. Notably, the I_D/I_G ratio is higher in GO1 compared to GO2, suggesting that GO1 has a higher concentration of defects or a greater degree of oxidation. Additionally, the broader full width at half maximum (FWHM) values of the D and G bands in GO1 (FWHM_D ≈ 49.55 cm^{-1}, FWHM_G ≈ 131 cm^{-1}) further support the presence of increased disorder when compared to GO2 (FWHM_D ≈ 37.92 cm^{-1}, FWHM_G ≈ 110 cm^{-1}). Moreover, the D′ shoulder band, a one-phonon defect-assisted mode, was observed more frequently across various laser spots in GO1, indicating inhomogeneous defect distribution or clustering. The D+D″ combination band also appears more intensely in GO1, reinforcing the conclusion of a higher defect density. Interestingly, an additional band at 643 cm^{-1}, which does not correspond to a known Raman mode of graphene oxide, appears only in GO1. This might be related to unique defect structures or contaminations and warrants further investigation.

5. Conclusion

Raman spectroscopy was used to characterize the structural disorder in two graphene oxide (GO) samples (Sample 1 and Sample 2), using a 633 nm excitation source. Both samples exhibited the characteristic D and G bands typically associated with disorder-induced and graphitic lattice vibrations, respectively. The intensity ratio of the D to G bands (I_D/I_G) serves as an indicator of the degree of disorder and the presence of sp^3-type defects in the GO lattice.

Sample 1 presented a D-band full width at half maximum (FWHM) of 49.55 cm^{-1}, while Sample 2 showed a narrower D-band FWHM of 37.92 cm^{-1}, suggesting relatively greater structural disorder in Sample 1. However, the calculated I_D/I_G ratios were 0.47 for Sample 1 and 0.59 for Sample 2. The lower I_D/I_G ratio in Sample 1 may reflect a higher degree of oxidation or a more amorphous carbon structure, in which the increase in disorder is no longer linearly correlated with the D-band intensity.

Additionally, spectral features such as a more prominent D′ shoulder band and a slightly stronger D + D″ composite band in Sample 1 further indicate a greater density of defect-related vibrational modes. A unique Raman feature at 643 cm^{-1}, observed only in Sample 1, may also be associated with defect-specific phonon modes or local vibrational anomalies.

In summary, while both samples exhibit disorder characteristics of oxidized graphene, the broader D-band and the appearance of additional defect-related modes in Sample 1 indicate more complex or diverse defect structures, despite its lower I_D/I_G ratio. These observations underline the importance of combining multiple Raman spectral features for a comprehensive assessment of GO quality and structural integrity.

6. References

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