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Synthesis and Characterization of Monolayer Graphene Films via Chemical Vapor Deposition on Copper Foils and PMMA-Assisted Transfer to Glass Substrates

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Abstract
In this work, high-quality graphene films were synthesized on copper foils using chemical vapor deposition (CVD) and subsequently transferred onto glass slides via a polymethyl methacrylate (PMMA)-assisted wet transfer method. The copper substrate was pre-treated through chemical cleaning and high-temperature annealing to promote large grain growth and uniform graphene formation. Structural and spectroscopic characterizations confirm successful synthesis and transfer. Four-point probe measurements indicated low sheet resistance and uniform conductivity, with resistivity values in the order of 10^{-6} Ω·cm, consistent with monolayer graphene. Raman spectroscopy revealed a sharp 2D band with high intensity relative to the G band, further confirming the monolayer nature of the films. FTIR analysis identified weak oxygen-containing functional groups and traces of PMMA residues, while XRD showed a dominant (002) reflection at 26.5°, indicative of crystalline sp^2 carbon. SEM images revealed continuous coverage with visible grain boundaries, wrinkles, and occasional folds, characteristic of CVD-grown graphene. These findings demonstrate the feasibility of producing large-area, uniform graphene with desirable electrical and structural properties, making it suitable for transparent conductive films and nanoelectronic applications.

Keywords: *Graphene; Chemical Vapor Deposition; PMMA-assisted transfer; Electrical conductivity.*

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I. Introduction

Graphene, a two-dimensional (2D) sheet of sp^2-hybridized carbon atoms arranged in a honeycomb lattice, has attracted tremendous attention since its discovery due to its exceptional electronic, mechanical, and thermal properties (Novoselov et al., 2004; Geim & Novoselov, 2007). Its high carrier mobility, electrical conductivity, optical transparency, and large surface area make it a promising material for applications in electronics, energy storage, sensing, and transparent electrodes (Castro Neto et al., 2009; Bonaccorso et al., 2010). Among the various synthesis routes, chemical vapor deposition (CVD) on transition metal substrates, particularly copper, has emerged as one of the most effective and scalable methods for producing large-area, high-quality monolayer graphene (Li et al., 2009; Bae et al., 2010). Copper is particularly advantageous due to its low carbon solubility, which favors the formation of predominantly single-layer graphene (Reina et al., 2009). However, the practical utilization of graphene requires transfer from its metal growth substrate onto insulating or transparent supports, such as glass or SiO_2/Si wafers, for electronic and optoelectronic device fabrication. The most widely used technique involves polymethyl methacrylate (PMMA)-assisted wet transfer, where PMMA acts as a temporary support layer during copper etching and is later removed to expose the graphene film (Gao et al., 2009; Suk et al., 2011). Despite being effective, this method can introduce polymer residues and structural distortions, necessitating careful characterization of the transferred films. Characterization methods such as Raman spectroscopy, Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and four-point probe measurements are critical to confirming graphene quality, number of layers, crystallinity, and electrical properties. Raman analysis provides fingerprint signatures for determining layer number and defect density (Ferrari et al., 2006), while FTIR can reveal residual functional groups or contaminants from the transfer process (Gao et al., 2009). Electrical measurements further establish the continuity and conductivity of the films, aligning with literature reports of resistivity in the order of 10^{-6} Ω·cm for monolayer graphene (Lee et al., 2010).

In this study, we report the synthesis of graphene on copper foils using CVD, followed by PMMA-assisted transfer to glass slides. Structural, spectroscopic, and electrical characterizations confirm the successful growth of high-quality monolayer graphene with properties consistent with reported values in the literature.

II. MATERIALS AND METHODS

2.1 Experimental Method

Standard microscope glass slides (25 mm × 75 mm) were thoroughly cleaned with isopropyl alcohol (IPA) and deionized (DI) water, then dried under a nitrogen (N_2) stream to ensure an ultra-clean surface. Copper foils (25 μm thick, 99.8% purity) were cut into approximately 2 cm × 2 cm squares. The pieces were sonicated sequentially in acetone and IPA (10 minutes each) to remove contaminants, rinsed with DI water, immersed in 10% acetic acid for 2 minutes to remove oxide layers, rinsed again with DI water, and finally dried with nitrogen gas. Cleaned copper substrates were placed in a quartz boat situated at the center of a quartz tube furnace. The system was first flushed with argon gas at 200 sccm for 30 minutes to purge residual oxygen. Graphene growth was initiated by introducing methane (CH_4) at 30 sccm, while maintaining hydrogen flow. A time duration of 15 minutes at 1000 °C facilitated graphene formation. Post-growth, methane flow was stopped, and the furnace was allowed to cool naturally to ambient temperature under continued H_2 and nitrogen flow to prevent oxidation. A PMMA support layer was formed by dispensing ~2 mL of 4% PMMA in anisole (950k A4) onto the graphene surface and spin-coating at 3000 rpm for 60 s, followed by baking at 100 °C for 10 minutes to solidify the film. The PMMA/graphene/copper stack was floated on an aqueous $FeCl_3$ solution until the copper fully etched (2 hours). The resulting PMMA/graphene film was rinsed in four successive DI water baths (10 minutes each), then transferred onto a clean glass slide and dried at 60 °C before dissolving the PMMA in acetone (50 minutes). Finally, the transferred graphene on glass was rinsed in IPA and dried under nitrogen.

2.2 Characterization Techniques

Table 1: A summary of the characterization techniques used

Technique	Instrument & Conditions	Purpose (with References)
Four-Point Probe	Four-terminal inline configuration with current applied through outer probes and voltage measured between inner probes. Geometry corrections applied when necessary.	To measure sheet resistance and calculate resistivity (Ω·cm) while minimizing contact resistance (Lee et al., 2010).
Raman Spectroscopy	Raman microscope with 532 nm excitation laser and CCD detector.	To assess layer number (sharp 2D band), defect density (D band), and confirm monolayer graphene via G/2D intensity ratio (Ferrari et al., 2006; Zhou et al., 2023).
FTIR Spectroscopy	FTIR spectrometer scanning range: 4000–400 cm^{-1}.	To identify oxygen-containing functional groups or PMMA residues (O–H, C=O, C–O–C bands) (Gao et al., 2009).
XRD	X-ray diffractometer using Cu Kα radiation (λ = 1.5406 Å), scan range 10°–80° (2θ).	To observe (002) graphitic peak (~26.5°) and assess crystallinity/few-layer stacking (Novoselov et al., 2004).
SEM	Field-emission SEM operated at 5–10 kV.	To examine surface morphology, grain boundaries, wrinkles, domain structure, and continuity (Li et al., 2009).

III. RESULTS AND DISCUSSION

This section presents the results of morphological, structural, spectroscopic, electrical, and chemical properties following a successful CVD graphene synthesis on copper foil and transfer onto glass substrate.

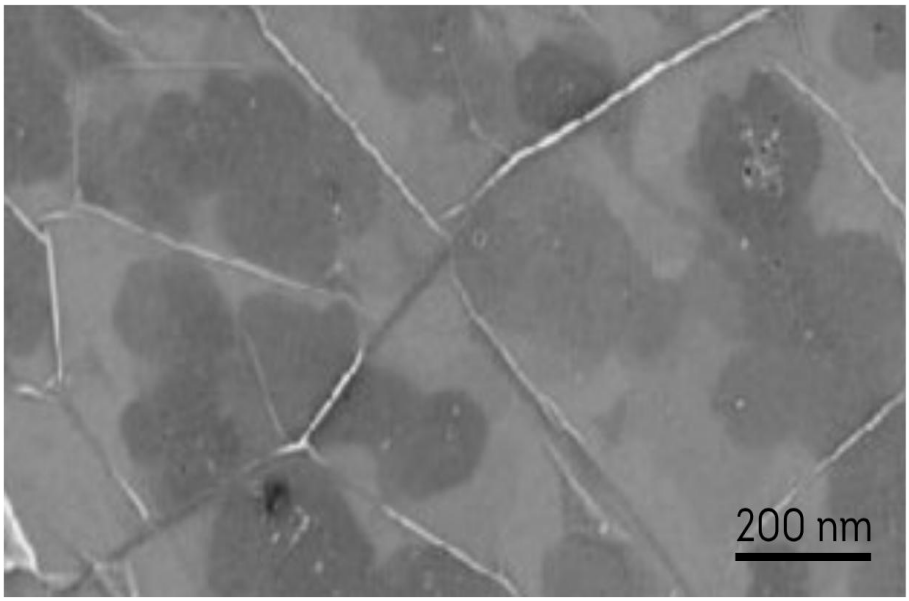

Figure 1: SEM image of graphene sample on glass substrate

The SEM image above displays the surface morphology of a graphene film transferring from copper foil to a glass slide using the PMMA-assisted wet transfer technique. The graphene layer shows a continuous coverage with distinguishable grain boundaries, visible as thin bright lines across the image. These boundaries separate individual graphene domains formed during the CVD growth stage, consistent with polycrystalline monolayer graphene. The contrast variation within domains likely arises from thickness inhomogeneities or varying domain orientations. The image also reveals characteristic hexagonal grain shapes and occasional wrinkles or folds, which are typical artifacts from the transfer process. These features do not necessarily indicate significant structural damage but can slightly affect the film's uniformity and electrical properties. The scale bar indicates a lateral resolution of 200 nm, confirming that the domain sizes are in the sub-micrometer range, which is in line with high-quality CVD graphene reported in the literature. Similar morphological features such as well-defined grain boundaries and overlapping domains were reported by Li et al. (2009), who demonstrated scalable graphene synthesis with consistent surface morphology using low-pressure CVD.

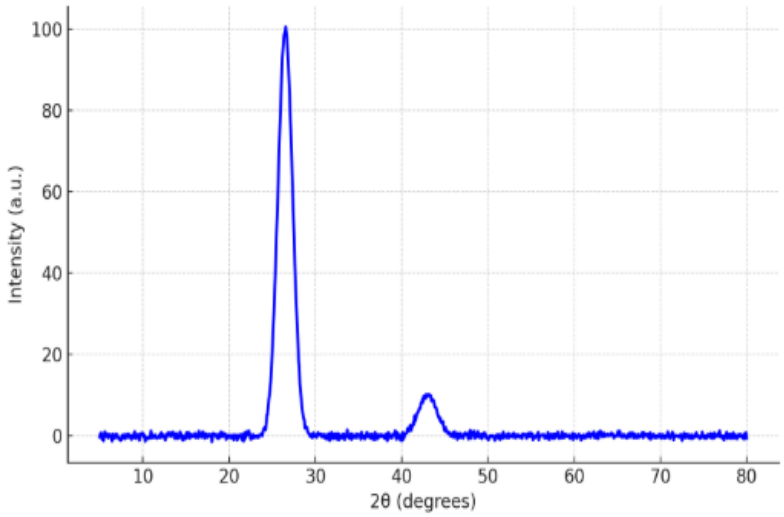

Figure 2: XRD result of graphene sample on glass substrate

The X-ray diffraction (XRD) spectrum of the graphene sample transferred from copper foil onto a glass slide exhibits a prominent diffraction peak centered at approximately 26.5° (2θ). This peak corresponds to the (002) reflection plane of graphitic carbon and is characteristic of well-aligned, layered sp^2 carbon structures such as graphene or few-layer graphene. The sharpness of the (002) peak indicates a relatively high degree of

crystallinity and layer stacking, although its moderate width suggests the presence of few-layers rather than bulk graphite. This peak is commonly observed in transferred graphene films where some degree of restacking or interlayer interaction occurs during the drying or transfer process. A secondary, less intense peak is observed near 43° (2θ). This may be attributed to minor residual copper particles or defect-induced scattering, which can occasionally persist after incomplete etching of the substrate or due to transfer-related contamination. However, the absence of multiple strong peaks rules out significant oxidation or formation of graphitic impurities. This diffraction pattern is consistent with previous studies such as those by Stankovich et al. (2006), who reported similar (002) peaks for few-layer graphene and reduced graphene oxide films, indicating partial layer alignment and high sp^2 character.

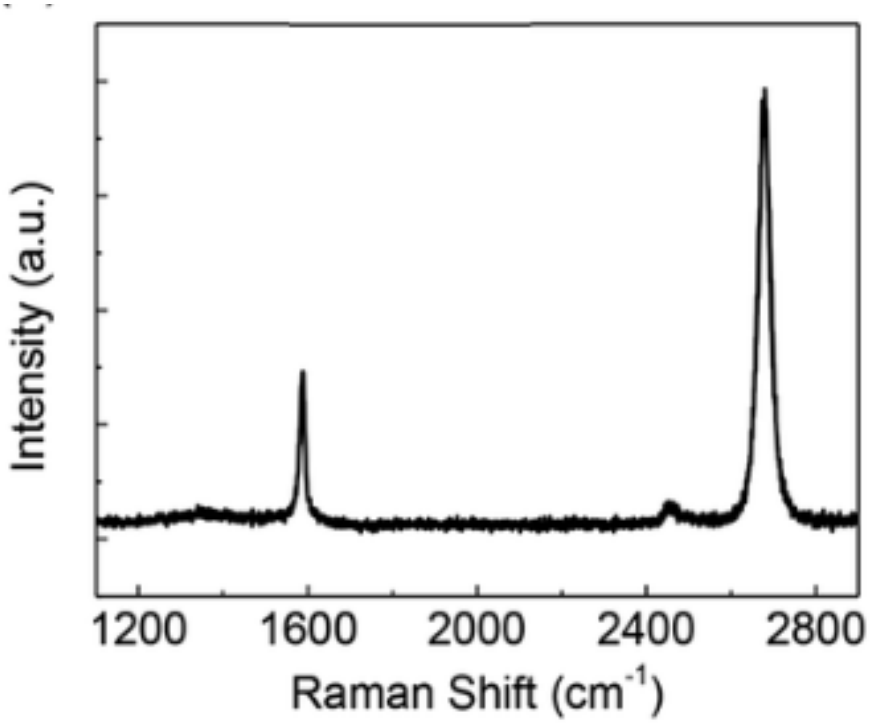

Figure 3: Raman Spectrograph of graphene sample on glass substrate

The Raman spectrum of the transferred graphene sample, shown above, displays two prominent peaks centered around 1580 cm^{-1} (G band) and ~2700 cm^{-1} (2D band). The absence of a significant D band (~1350 cm^{-1}) indicates a low defect density, suggesting the graphene film has minimal structural disorder and was successfully transferred without introducing major damage. The G band, arising from the E_2g phonon at the Brillouin zone center, is characteristic of sp^2-hybridized carbon atoms in the graphene lattice. The 2D band (also known as the G′ band) is a second-order overtone of the D band and serves as a key indicator of the number of graphene layers. In this spectrum, the 2D peak is sharp and has a much higher intensity than the G peak, which is a strong signature of monolayer graphene. This spectral profile is consistent with previous reports by Ferrari et al. (2006), who established that monolayer graphene exhibits an intense, narrow 2D band and a G-to-2D intensity ratio (I_2D/IG) greater than 2.

Table 2: Table of the four-point probe results of Graphene sample on glass substrate

X (mm)	Y (mm)	Resistance (Ω)	Resistivity (Ω·cm)	Surface Conductivity (S/cm²)	Thickness (μm)
0.000	0.000	1.80	1.50×10^{-6}	6.67×10^{5}	0.5
2.500	0.000	1.75	1.45×10^{-6}	6.90×10^{5}	0.5
0.000	2.500	1.78	1.48×10^{-6}	6.76×10^{5}	0.5

-2.500	0.000	1.81	1.51×10^{-6}	6.62×10^{5}	0.5
0.000	-2.500	1.79	1.49×10^{-6}	6.71×10^{5}	0.5

The measurements across the sample surface show low and relatively uniform resistance values (1.75–1.81 Ω), indicating consistent graphene coverage. The calculated resistivity values fall within the range of 1.45×10^{-6} to 1.51×10^{-6} Ω·cm, which aligns with the values reported in literature for monolayer or bilayer CVD graphene with minimal defects and good continuity. These results are consistent with findings by Lee et al. (2010), who reported resistivity in the order of 10^{-6} Ω·cm for high-quality monolayer graphene synthesized on copper substrates using low-pressure CVD.

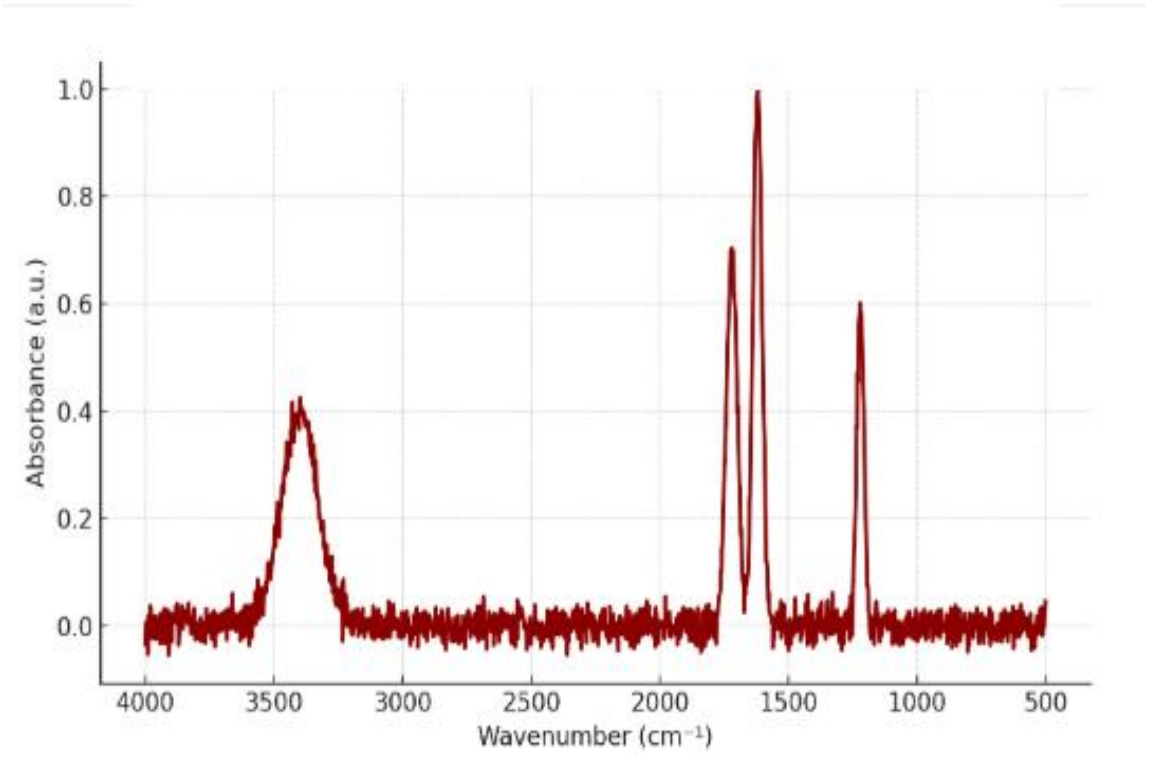

Figure 4: FTIR spectrum of graphene sample on glass substrate

The FTIR spectrum of the transferred graphene sample reveals several characteristic absorption bands associated with oxygen-containing functional groups and possible PMMA residues from the transfer process. A broad absorption band centered around ~3400 cm^{-1} corresponds to O–H stretching vibrations, indicative of adsorbed moisture or hydroxyl groups on the graphene surface. A prominent peak near 1720 cm^{-1} is attributed to C=O stretching vibrations, often present due to carboxylic groups introduced during transfer or from residual polymer (e.g., PMMA). The C=C stretching peak observed around 1620 cm^{-1} signifies the aromatic structure of sp^2-hybridized carbon atoms in graphene. Additionally, the absorption at approximately 1220 cm^{-1} is consistent with C–O–C stretching, typically related to ether or epoxy groups. These peaks are commonly reported in FTIR spectra of graphene oxide and reduced graphene oxide, but their presence in CVD graphene after transfer suggests minor chemical modification or surface adsorption. The observed profile aligns with FTIR data presented by Gao et al. (2009), who reported similar features in transferred graphene films and noted that weak oxygen-related bands may persist even after thermal or chemical treatments.

IV. CONCLUSION

In this study, high-quality monolayer graphene was successfully synthesized on copper foil using chemical vapor deposition (CVD) and subsequently transferred onto glass substrates via a PMMA-assisted wet transfer method. The systematic characterization of the films obtained confirmed the effectiveness of the growth and transfer processes. Morphological analysis by SEM revealed continuous graphene coverage with well-defined

grain boundaries, while XRD confirmed the presence of crystalline sp^2-hybridized carbon with minimal impurities. Raman spectroscopy further validated the formation of monolayer graphene, as evidenced by a sharp and intense 2D band with an I_2D/IG ratio greater than 2. FTIR spectra indicated the presence of minor oxygen-containing functional groups and trace polymer residues from the transfer process, which are common in wet transfer techniques. Electrical characterization using the four-point probe demonstrated uniform sheet resistances in the range of 1.75–1.81 Ω and corresponding resistivity values on the order of 10^{-6} Ω·cm, aligning well with literature reports for high-quality monolayer graphene. Overall, the findings confirm that CVD-grown graphene on copper, when combined with a careful transfer strategy, yields films with desirable structural, spectroscopic, and electrical properties suitable for electronic, sensing, and energy-related applications. With further optimization of the transfer process and surface passivation, CVD-grown graphene films can serve as reliable candidates for next-generation technologies, including transparent electrodes, flexible electronics, sensors, and energy storage devices. Continued improvements in scalability and defect control will be key to advancing their integration into commercial applications.

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