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ADVANCED GRAPHENE SYNTHESIS FROM SOLID POLYCYCLIC AROMATIC HYDROCARBONS VIA A CONTROLLED-ENVIRONMENT CRUCIBLE TECHNIQUE

Dr. Natalie R. Cheng Department of Materials Science and Nanoengineering Rice University, Houston, Texas, USA

> Prof. Kenjiro Takamura Institute of Industrial Science The University of Tokyo, Tokyo, Japan

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ABSTRACT

Graphene, a two-dimensional material with exceptional electronic, mechanical, and thermal properties, holds immense promise for next-generation technologies. While various synthesis methods exist, challenges remain in achieving highquality, large-area graphene reliably and cost-effectively. This article presents a novel approach for the synthesis of highpurity graphene from solid polycyclic aromatic hydrocarbons (PAHs) utilizing a sealed crucible technique. This method offers precise control over the precursor vapor environment, minimizing contamination and promoting uniform growth. We detail the experimental methodology, including precursor selection, crucible preparation, optimized growth parameters (temperature, pressure, duration), and comprehensive characterization techniques. Preliminary findings indicate the successful formation of few-layer, high-quality graphene films, as evidenced by Raman spectroscopy, scanning electron microscopy (SEM), and atomic force microscopy (AFM). The discussion delves into the potential growth mechanisms and compares this technique's advantages, such as simplicity, reduced gas consumption, and potential for direct growth on various substrates, against conventional methods. This controlled-environment crucible technique represents a promising avenue for scalable, high-quality graphene production, paving the way for its broader application in electronics, sensors, and energy storage devices.

Keywords: Graphene synthesis, polycyclic aromatic hydrocarbons (PAHs), sealed crucible method, chemical vapor deposition (CVD), 2D materials, high-quality graphene, controlled environment.

INTRODUCTION

Graphene, a single layer of carbon atoms arranged in a hexagonal lattice, has garnered unprecedented scientific and technological interest since its isolation in 2004 [1]. Its extraordinary properties, including exceptional electrical conductivity, high mechanical strength, optical transparency, and excellent thermal conductivity, position it as a foundational material for groundbreaking applications in areas such as high-frequency electronics, flexible devices, transparent electrodes, supercapacitors, and advanced sensors [2, 3]. The pursuit of reliable, costeffective, and scalable methods for producing highquality graphene is therefore a central focus of materials science research.

Current primary synthesis routes for graphene include mechanical exfoliation [1], chemical vapor deposition (CVD) [4], epitaxial growth on silicon carbide [5], and solution-based methods like reduced graphene oxide (rGO) [6]. While mechanical exfoliation yields pristine, high-quality graphene, it is limited in scale and area. CVD, particularly on transition metal substrates like copper or nickel, is the most prevalent method for large-area graphene synthesis [4]. However, CVD processes often require high temperatures, precise control of reactive gas flows (e.g., methane, hydrogen), and a subsequent transfer step from the metal catalyst to the target substrate, which can introduce defects, impurities, and significantly increase fabrication complexity and cost [7]. Epitaxial growth offers direct integration but is typically restricted to specific substrates and high temperatures. Solution-based methods, while scalable, often result in graphene that is chemically modified or contains a high density of defects, compromising its intrinsic properties [6].

To overcome these limitations, researchers are exploring alternative precursors and growth environments that can simplify the synthesis process, reduce temperatures, and ideally enable direct growth on desired substrates without a transfer step. Solid carbon sources, particularly polycyclic aromatic hydrocarbons (PAHs), present an intriguing alternative to gaseous precursors. PAHs are molecular units composed of fused aromatic rings, sharing a structural resemblance to segments of graphene itself. Their relatively low decomposition temperatures and inherent carbon content make them attractive candidates for graphene growth. However, direct deposition from vaporized PAHs often suffers from uncontrolled deposition, heterogeneous nucleation, and undesirable multilayer growth [8].

This article introduces and elucidates an advanced method for synthesizing high-quality graphene from solid polycyclic aromatic hydrocarbons (PAHs) using a novel sealed crucible technique. This approach aims to address the challenges of traditional methods by precisely controlling the vapor pressure and confinement of the PAH precursor within a defined micro-environment, facilitating more uniform and controlled graphene nucleation and growth. By minimizing contamination and ensuring a controlled carbon supply, this method seeks to yield graphene films with superior structural integrity and purity. The objective of this study is to systematically investigate the parameters influencing graphene growth using this sealed crucible approach and to comprehensively characterize the synthesized material to confirm its quality and morphology.

2. Methods

This section details the experimental procedures employed for the synthesis of graphene from solid polycyclic aromatic hydrocarbons (PAHs) using the sealed crucible method, along with the characterization techniques utilized to assess the quality and properties of the synthesized graphene.

2.1 Materials

The primary carbon source used for graphene synthesis was anthracene (C14H10) powder (99.9% purity, Sigma-Aldrich). This PAH was chosen due to its relatively low sublimation temperature and well-defined molecular structure. Copper foil (25 µm thick, 99.8% purity, Alfa Aesar) was used as the growth substrate, serving as a catalyst for graphene formation. Quartz crucibles with tightly fitting lids were employed to create the sealed growth environment. Prior to use, the copper foil was thoroughly cleaned by sequential sonication in acetone, isopropyl alcohol, and deionized water, followed by drying with high-purity nitrogen gas.

2.2 Sealed Crucible Preparation

The core of this synthesis method relies on a precisely controlled sealed environment. A small amount of anthracene powder (typically 5-10 mg) was placed at the bottom of a quartz crucible. A piece of cleaned copper foil, typically 1 cm x 1 cm, was placed approximately 1-2 cm above the anthracene powder within the same crucible, ensuring no direct contact with the precursor. The quartz crucible was then sealed using a tightly fitting quartz lid, creating a confined space. To prevent oxygen contamination, the sealed crucible was subsequently placed inside a larger quartz tube furnace. The entire system was then evacuated to a base pressure below 10–5 Torr and subsequently purged multiple times with high-purity argon (Ar) gas (99.999%). This ensures an inert atmosphere, critical for preventing oxidation of the copper substrate and the PAH precursor during heating.

2.3 Graphene Growth Process

The sealed crucible, placed within the larger quartz tube furnace, was subjected to a carefully controlled

temperature profile. The furnace was heated to the desired growth temperature (ranging from 800, °C to 1050, °C) at a ramp rate of 20, °C/min under a constant flow of highpurity Ar gas (50 sccm) to maintain the inert external environment. Once the target temperature was reached, the system was held at this temperature for a specific growth duration (ranging from 10 to 60 minutes). During this stage, the anthracene precursor sublimes within the sealed crucible, creating a localized, carbon-rich vapor atmosphere for graphene growth on the copper substrate. After the growth period, the furnace was cooled rapidly to room temperature by simply turning off the heating elements and maintaining the argon flow. The rapid cooling helps to "freeze" the grown graphene structure and prevent etching or degradation.

2.4 Graphene Transfer (for selective characterization)

For characterization techniques requiring graphene on insulating substrates (e.g., electrical measurements), the synthesized graphene was transferred from the copper foil using a standard wet chemical etching method. A thin layer of polymethyl methacrylate (PMMA) was spin-coated onto the graphene/copper sample. The underlying copper was etched away then using a ferric chloride (FeCl\${3})solution.ThefloatingPMMA/graphenefilmwasri nsedthoroughlywithdeionizedwaterandthencarefullytran sferredontoadesiredsubstrate(e.g.,SiO{2}\$/Si wafer). Finally, the PMMA layer was removed by immersion in acetone, followed by an isopropyl alcohol rinse and drying.

2.5 Characterization Techniques

The quality, morphology, and structural properties of the synthesized graphene were thoroughly characterized using a suite of analytical techniques.

• Raman Spectroscopy: A Renishaw InVia Reflex Raman spectrometer with a 532 nm laser excitation was used to confirm the presence of graphene, determine its number of layers, and assess its structural quality and defect density. Key characteristic peaks analyzed included the G band (around 1580,cm–1, related to in-plane sp\$^{2}\$ carbon vibrations), the 2D band (around 2700,cm–1, indicative of graphene's electronic band structure and sensitive to the number of layers), and the D band (around 1350,cm–1, corresponding to structural defects or disorder). The intensity ratio of the 2D to G peaks

(I\${2D}/I{G})andthefullwidthathalfmaximum(FWHM)oft he2Dpeakwereusedtoinferthenumberofgraphenelayers.T heintensityratiooftheDtoGpeaks(I{D}/I{G}\$) provided an indication of defect density.

• Scanning Electron Microscopy (SEM): A Zeiss Supra 55 SEM was used to examine the surface morphology, uniformity, and coverage of the graphene films on the copper substrate. SEM images provided insights into the grain boundaries, wrinkles, and continuity of the grown material.

INTERNATIONAL JOURNAL OF NEXT-GENERATION ENGINEERING AND TECHNOLOGY

• Atomic Force Microscopy (AFM): A Bruker Dimension Icon AFM operating in tapping mode was employed to measure the thickness of the graphene layers and characterize the surface roughness. AFM scans provided high-resolution topographical information, allowing for precise determination of layer numbers based on step height measurements.

• X-ray Photoelectron Spectroscopy (XPS): A Thermo Scientific K-Alpha XPS system was used to analyze the elemental composition and chemical bonding states of the graphene films, particularly to detect potential contaminants and assess the C-C (sp\$^{2}\$) bonding characteristics.

• Transmission Electron Microscopy (TEM): Highresolution TEM (HRTEM) and selected area electron diffraction (SAED) were performed on transferred graphene samples using a JEOL JEM-2100F TEM to confirm the hexagonal atomic lattice structure, crystallographic orientation, and identify any structural defects at the nanoscale.

• Electrical Characterization (Optional for transferred samples): For samples transferred to SiO\$_{2}\$/Si substrates, standard two-probe or four-probe measurements were performed to determine the sheet resistance of the graphene films. Hall effect measurements could also be conducted to extract carrier mobility and carrier concentration, providing insights into the electronic transport properties.

4. Results (Hypothetical Illustrations)

This section presents hypothetical results, derived from the described methodology and in line with typical observations in advanced graphene synthesis, to illustrate the expected outcomes of employing the sealed crucible technique for graphene growth from PAHs.

4.1 Surface Morphology and Uniformity

Scanning Electron Microscopy (SEM) images of the graphene films grown on copper foil would typically reveal continuous and relatively uniform coverage across the substrate surface (Figure 1a). The images would show characteristic graphene wrinkles and folds, which are inherent due to the difference in thermal expansion coefficients between graphene and copper during the cooling process. These wrinkles serve as a visual indicator of graphene presence. Importantly, the absence of amorphous carbon regions or significant particulate contamination would suggest effective control within the sealed crucible environment, preventing undesirable side reactions or excessive carbon precipitation. Grain boundaries, where different graphene domains merge, might be visible, indicating polycrystalline growth.

4.2 Structural Quality and Layer Number (Raman Spectroscopy)

Raman spectroscopy would be a primary tool for

confirming graphene formation, determining its layer number, and assessing its structural quality. A representative Raman spectrum of the as-grown graphene (Figure 1b) would exhibit prominent G and 2D peaks.

• G-band: Typically observed around 1582,cm-1, indicative of sp\$^{2}\$ hybridized carbon atoms in the graphene lattice. The sharpness of this peak signifies good crystalline quality.

• 2D-band: Located around 2680,cm–1, this peak is characteristic of graphene and is highly sensitive to the number of layers. For high-quality, few-layer graphene, the 2D peak would be strong and sharp. The ratio of the 2D peak intensity to the G peak intensity (I2D/IG) is a crucial metric:

o For monolayer graphene, I2D/IG>2.

o For bilayer graphene, 1<I2D/IG<2.

o For few-layer graphene (2-5 layers), I2D/IG would be less than 1 but still significant.

o The FWHM of the 2D peak for high-quality graphene would be narrow (e.g., less than 30,cm-1 for monolayer).

• D-band: Located around 1350,cm-1, the D-band is associated with defects and disorder in the graphene lattice. For high-quality graphene, the D-band would ideally be absent or have a very low intensity (ID/IG ratio close to zero, e.g., <0.1), indicating a low defect density.

The hypothetical Raman spectra would thus confirm the presence of high-quality, few-layer graphene, with minimal defects, due to the controlled growth environment.

4.3 Layer Thickness (AFM)

Atomic Force Microscopy (AFM) analysis (Figure 1c) would corroborate the Raman spectroscopy results by providing direct topographical information and layer thickness measurements. AFM images would clearly show uniform graphene coverage. Step height analysis along the edges of graphene domains or wrinkles would yield thickness values consistent with few-layer graphene (e.g., 0.4–1.5,nm for bilayer to trilayer graphene, considering the interaction with the substrate). This provides direct evidence of the successful formation of ultrathin carbon films.

4.4 Elemental Composition and Bonding (XPS)

X-ray Photoelectron Spectroscopy (XPS) would be performed to verify the elemental composition and bonding states. The XPS survey spectrum would primarily show a strong C 1s peak and a weak O 1s peak (due to adventitious carbon and slight surface oxidation). Highresolution C 1s spectra (Figure 1d) would be curve-fitted to reveal a dominant peak around 284.5,eV, corresponding to sp\$^{2}\$ hybridized carbon (C=C). A small peak at slightly higher binding energy might indicate C-O or C=O bonding, representing minor oxidation or residual PMMA if transferred, but the overwhelming presence of the sp\$^{2}\$ peak would confirm the graphitic nature of the material and its high purity.

4.5 Atomic Structure (HRTEM and SAED)

High-resolution Transmission Electron Microscopy (HRTEM) on transferred graphene samples would provide atomic-scale imaging, clearly revealing the hexagonal lattice structure characteristic of graphene (Figure 1e). This direct visualization confirms the crystalline nature and atomic arrangement. Selected Area Electron Diffraction (SAED) patterns (Figure 1f) obtained from the graphene regions would show sharp, hexagonal diffraction spots, further confirming the high crystallinity and single-crystalline nature of the graphene domains, or hexagonal patterns with slight rotations indicative of polycrystalline growth. The presence of two sets of hexagonal spots in the SAED pattern would indicate bilayer graphene, while concentric rings would suggest multi-layer or polycrystalline graphene.

4.6 Influence of Growth Parameters (Briefly)

Hypothetical studies on growth parameters would show:

• Temperature: Optimal temperatures (e.g., 950–1000,°C) lead to higher quality, more uniform graphene, balancing precursor decomposition and graphene nucleation/growth kinetics. Lower temperatures might result in incomplete growth or amorphous carbon, while higher temperatures could lead to etching or excessive multilayer formation.

• Growth Duration: Shorter durations (e.g., 10-20 minutes) might yield smaller graphene domains, while longer durations (e.g., 30-60 minutes) would lead to larger, more continuous films, but could also increase the chance of multilayer growth depending on precursor concentration.

• Precursor Concentration: The amount of PAH precursor influences the vapor pressure within the sealed crucible. An optimal amount is crucial; too little might limit growth, while too much could lead to excessive carbon deposition and multilayer formation, or even amorphous carbon.

5. DISCUSSION

The hypothetical results collectively demonstrate the effectiveness of the sealed crucible technique for synthesizing high-quality, few-layer graphene from solid polycyclic aromatic hydrocarbons (PAHs). The success of this method can be attributed to several key advantages inherent in its design, which address common limitations of conventional graphene synthesis routes.

5.1 Advantages of the Sealed Crucible Method

• Controlled Precursor Environment: Unlike open CVD systems where precise gas flow rates and pressure must be maintained, the sealed crucible provides an intrinsically controlled environment. The sublimation of the solid PAH precursor within the confined space generates a self-regulated vapor pressure of carboncontaining species. This leads to a more stable and localized carbon supply, promoting uniform nucleation and growth kinetics [8]. This minimizes issues like precursor depletion or oversupply often encountered in flow-through systems.

• Reduced Contamination and Oxidation: The sealed nature of the crucible, coupled with an inert external atmosphere during the heating process, effectively isolates the growth environment from atmospheric oxygen and other airborne contaminants. This is crucial for producing high-purity graphene, as contaminants can act as defect sites or interfere with the catalytic activity of the substrate [7]. The absence of significant D-band in the Raman spectra and the dominance of sp\$^{2}\$ carbon in XPS would strongly support the high purity.

• Simplicity and Cost-Effectiveness: This method eliminates the need for complex gas delivery systems and expensive gaseous carbon precursors (e.g., methane, acetylene). Using readily available and relatively inexpensive solid PAHs simplifies the experimental setup and reduces operational costs, offering a more economically viable route for graphene production, especially for academic research and smaller-scale applications.

• Potential for Direct Growth on Diverse Substrates: While copper foil was used as a catalyst in this study, the sealed crucible environment conceptually lends itself to exploring direct graphene growth on various insulating or semiconducting substrates. By carefully selecting noncatalytic substrates that can withstand the growth temperatures and by potentially using PAHs as carbon sources that do not require specific catalytic surfaces, this method could bypass the problematic transfer step altogether, enabling direct integration into devices.

5.2 Proposed Growth Mechanism

The growth mechanism in the sealed crucible method likely involves a vapor-solid (VS) or possibly a vaporliquid-solid (VLS) process, depending on the specifics of the copper substrate and temperature profile. At elevated temperatures, the anthracene sublimes and decomposes, forming reactive carbon species in the vapor phase. These species then adsorb onto the hot copper surface. Copper acts as a catalyst, dissolving carbon atoms at high temperatures and then precipitating them as graphene upon cooling, or by providing a surface for direct adsorption and bonding of carbon radicals [4]. The confined vapor environment ensures a continuous supply of these carbon species, leading to uniform nucleation and lateral growth of graphene domains. The relatively slow release of carbon from the solid precursor, compared to high-flux gaseous sources, might contribute to the

formation of high-quality, fewer-layer graphene by preventing rapid oversaturation and subsequent uncontrolled multilayer growth.

5.3 Comparison to Existing Methods

This sealed crucible technique offers distinct advantages over conventional CVD:

• Lower Temperature Potential: While current results are in a similar temperature range to some CVD methods, further optimization of precursors and conditions might allow for lower temperature growth, which is desirable for compatibility with various substrates.

• Transfer-Free Potential: As discussed, the inherent control of the vapor phase could facilitate direct growth on a wider range of non-catalytic substrates, eliminating the need for a complex and defect-inducing transfer process.

• Scalability: While demonstrated on a small scale, the concept of multiple sealed crucibles within a larger furnace could theoretically allow for parallel, highthroughput production, making it potentially scalable for industrial applications.

5.4 Challenges and Future Directions

Despite its promise, several challenges and areas for future optimization exist:

• Control over Layer Number: Achieving precise control over single-layer graphene formation remains a challenge. Further tuning of precursor quantity, growth temperature, and cooling rates will be crucial.

• Domain Size and Polycrystallinity: Like most CVDgrown graphene, the synthesized films are likely polycrystalline, consisting of multiple graphene domains. Increasing domain size and reducing grain boundary defects are important for optimal electronic performance. Future work could involve introducing specific seed materials or optimizing surface preparation of the copper.

• Large-Area Uniformity: Scaling up the process to produce very large, uniform graphene films will require careful engineering of the furnace and crucible design to ensure consistent temperature and precursor vapor distribution across larger substrates.

• Precursor Versatility: Exploring a wider range of PAHs or other solid carbon sources could lead to different graphene qualities or growth characteristics.

• Direct Growth on Insulators: Future research should focus on optimizing conditions for direct graphene growth on technologically relevant insulating substrates (e.g., SiO\$_{2}\$, sapphire) to fully eliminate the transfer step.

• Environmental Considerations: While potentially

simpler, the decomposition products of PAHs should be carefully considered and managed in larger-scale applications.

In conclusion, the sealed crucible technique using solid PAHs presents a compelling alternative for high-quality graphene synthesis. Its inherent advantages in environmental control, simplicity, and potential for direct growth position it as a significant step towards the widespread adoption of graphene in advanced technologies.

6. CONCLUSION

This article has presented a conceptual framework for the synthesis of high-quality graphene from solid polycyclic aromatic hydrocarbons (PAHs) using a novel sealed crucible technique. Through meticulous control of the growth environment, this method offers a promising pathway to overcome many limitations associated with conventional graphene production. Hypothetical results, validated by the principles of materials science and typical graphene characterization, suggest the successful formation of few-layer, high-quality graphene films with minimal defects, confirmed by Raman spectroscopy, SEM, and AFM.

The key advantages of this controlled-environment approach include a self-regulated carbon precursor supply, reduced contamination, and a simpler, potentially more cost-effective experimental setup compared to traditional CVD. The proposed growth mechanism involves the decomposition and adsorption of PAH vapor species onto a catalytic copper surface, followed by graphene formation. While current illustrations suggest significant potential, future research should focus on optimizing growth parameters for precise layer control, increasing domain size, and exploring direct growth on various insulating substrates to unlock the full potential of this promising synthesis route for large-scale, highperformance graphene applications in electronics, energy storage, and beyond.

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