



Going Ballistic: High Quality Graphene Growth for Graphene Based Hot Electron Transistor

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Executive Summary

[Author: FC; Editor: NW, BT] Next-generation semiconductor devices, which will power advanced technologies like 6G telecommunications and instrumentation detection, require the incorporation of advanced materials to bring them to fruition. One such material is graphene, consisting of just one atomic layer of a carbon network. Its exceptional electrical and mechanical properties can increase the efficiency of a hot electron transistor (HET), a semiconductor based logical device. However, current methods to fabricate graphene on the substrates introduce contaminants during mechanical transport and exfoliation, or require elevated processing temperatures that degrade the underlying substrate and dielectric. To circumvent these constraints, we are developing a method that coats a substrate with a polymer through a dip-coating process and then graphitizes it by thermal treatment. The dip coating process allows for a thin polymer film to be deposited on a substrate without mechanical transport and exfoliation. Thermal treatment is used to determine if the polymer is a suitable graphene precursor, which will facilitate future research into replacing this thermal process with a plasma enhanced chemical vapor deposition (PECVD), overcoming the thermal hurdle.

While many polymers, such as polymethyl methacrylate (PMMA), can become graphitic at higher temperatures, we hypothesize that selecting a polymer with a molecular structure most similar to graphene will facilitate the conversion process at lower temperatures. Polyimides (PI), with a high density of sp² carbons and aromaticity, fits this description and became our polymer of choice. To confirm our hypothesis, we exposed PI and PMMA to high temperatures and analyzed their degree of graphitization using Raman spectroscopy, the standard analytical technique for graphene characterization. Our results confirmed that PI was indeed a better precursor than PMMA for graphitization.

Achieving a single atomic layer of graphene remains a significant challenge, as the thinnest film thickness we have achieved after thermal treatment is still greater than 10 monolayers of graphene's

thickness. To overcome the limitations of dip coating that we have observed in this project, we propose exploring other alternatives, such as spin coating or an ultrasonic spray system. Further consultation and research will be necessary to determine the most effective approach.

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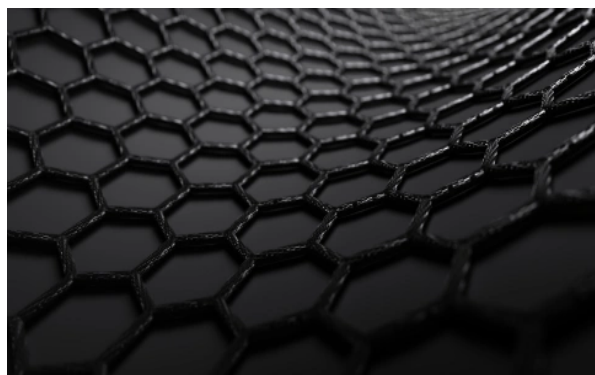
Section I. Critical Business & Contextual Literature Reviews

1a. Introduction

[Author: NW; Editor: FC] As the semiconductor industry advances towards enabling enhanced detection, sensitivity, and amplified data transmission technologies so does the search for new materials that can be controlled by electric fields (Novoselov et al. 2004) - graphene, wholly carbon based, is such a material that is tuneable to possess near zero electrical resistances during electron conduction or not conduct electricity at all. The successful incorporation of graphene in modern electronics is a next step in transitioning from 5G and into the potential of 6G and THz technologies. Terahertz electromagnetic radiation, whose wavelength encompasses the lower end of radio waves and higher end of light waves (infra-red), can penetrate opaque materials allowing for enhanced sensitivity detection. The depth of the revealed information relayed through THz wavelengths has major implications in illuminating in richer detail molecular/chemical processes in biology, medical spectroscopies, food and agriculture inspections as well as homeland security and screening. While set in theory, actualizing such potential starts with introducing advanced materials into the composition of a semiconductor chip.

III-N semiconductor devices, such as Hot Electron Transistors (HETs), whose emitter base is composed of a highly tunable electronically conductive material are ready for an upgrade. HETs with emitter bases composed of organic conductors and carbon nanotubes have shown promise, although metals still reign supreme. However, using metals would require atomically thin films that tend to be thermodynamically unstable as metals prefer coalescing, thus leading to discontinuous films thereby impeding tuneable electric field effects. Graphene, one to a few atomic layers of graphite and a naturally occurring material whose molecular structure can be visualized as a honeycomb sheet of carbon atoms (Figure 1), has broken this hindrance. This repeating hexagonal, planar, carbon structure

(a)



(b)

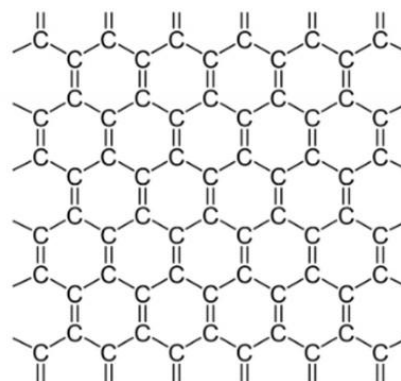


Figure 1. The atomic structure of graphene. (a) The microscopic rendition of a graphene nanosheet. (b) The chemical structure of graphene where c represents the carbon atom in the network.

has excellent mechanical properties which makes it flexible and strong as well as possessing unique electrical properties - one such electrical property is its ballistic transport mechanism that allows for electrons to flow with near zero resistance even at extremely low temperatures, as previously mentioned (Mayorov et al. 11). Additionally, it is also an excellent thermal conductor. These properties make graphene one of the most advanced materials continuously under study, research, and development given its vast implications for future device applications (Son and Ham 2017).

As such, graphene incorporation in HETs shows profound technological promise. This type of transistor operates by exploiting the phenomenon of electron tunneling effect, or hot carrier injection, which occurs when electrons in a semiconductor material gain a large amount of kinetic energy due to a high electric field or high temperature. This results in the injection of "hot" electrons into the conduction band of the semiconductor material - simply put, this property serves to amplify a signal. Given this enhancement in signal to noise property of HETs, they are then poised to replace many bipolar transistors due to their superior gain and higher cut-off frequencies. Given the ultrathin and high conductivity of graphene, it is then crucial and of substantial commercial interest to grow a monolayer of graphene on a III-N semiconductor.

While graphene fabrication on semiconductors has already been established, methods of fabrication either introduce impurities and more importantly, require high temperatures which begins to degrade the underlying semiconductor and dielectric. Therefore, our project aims to develop a method for fabricating a component of a HET by growing a monolayer of graphene directly on a wide-band gap semiconductor such as Gallium Nitride (GaN) and at the lowest temperatures possible. Moreover, successfully achieving this has implications for transferring the process into futuristic technologies, including flexible electronics, separation membranes, or biomedical skin-like devices.

1b. Existing Solutions

[Author: BT; Editor: FC] As a result of the significant demand for semiconducting graphene, numerous technical solutions have been discovered to achieve monolayer growth. The most archaic and widely recognized method is mechanical exfoliation of pre-existing graphite, which utilizes scotch

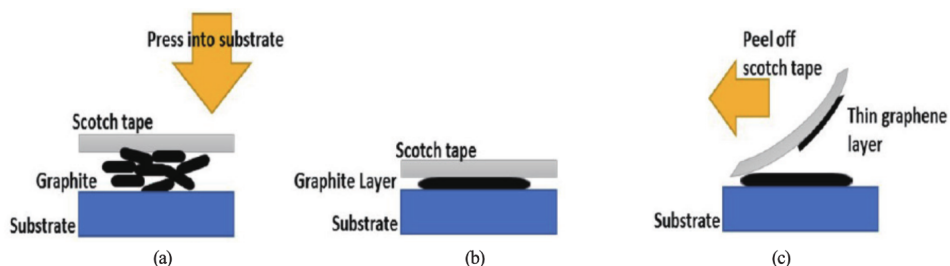


Figure 2. The process of peeling is repeated from steps (a) to (c).

tape to repetitively peel a sheet of graphite until it is reduced to a thin layer of graphene, shown in Figure 2 (Demon, 2022). This method offers the benefit of simplicity, making it easy for lab researchers to operate. However, due to the pressure differences in the peeling process, the size, shape, and thickness of the resulting film can be difficult to control, which makes this process undesirable in the commercial space.

Graphene can also be synthesized by exposing a carbon source to elevated temperatures, generally greater than 1200°C. Temperatures for graphene growth are also dependent on the ambient pressure, with high pressures being more conducive. Carbon sources are typically polymeric in nature and it has been shown that polymethyl methacrylate (PMMA) can graphitize at elevated temperatures. Not exclusive to PMMA, other polymers can also be graphitized at elevated temperatures depending on multiple factors ranging from their elemental composition and bonding as well their packaging geometries. Nonetheless, graphitization via thermal annealing requires elevated temperatures. Due to the challenges of producing high-quality graphene films and at lower temperatures, more advanced and intricate techniques are required.

Chemical vapor deposition (CVD) has emerged as a popular approach for depositing large-area, high-quality graphene films on substrates. This method, shown in Figure 3, involves

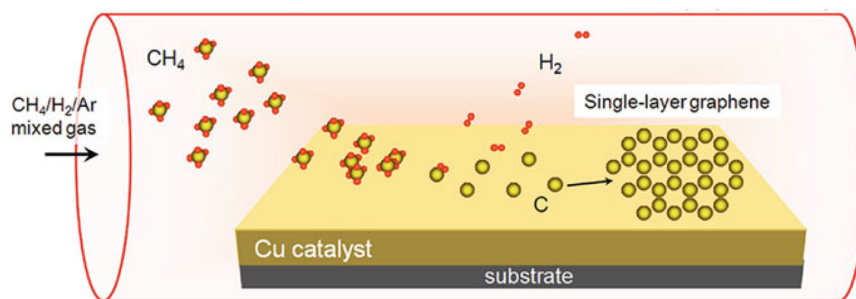


Figure 3. CVD graphene growth procedure.

introducing a flow of hydrocarbon precursor and an inert gas into a heating chamber, where the precursor undergoes a gas-phase reaction and transports itself to the surface of the substrate, where it diffuses. Carbon atoms mostly adsorb onto the surface while hydrogen atoms desorb from it, and the carbon atoms then rearrange themselves under high thermal conditions. Any remaining precursor gas reacts and creates a byproduct (Matsumoto 2015, Ch.1 pg.5). Normally, a catalyst, such as copper, is added to the substrate to lower the growth temperature inside the chamber to approximately 900 to 1080 °C during the CVD process of graphene (Matsumoto 2015, Ch.1 pg.5). Despite the catalyst's impact, the process still requires a significant thermal budget, which can increase production costs and impose

restrictions on the selection of substrate materials. Therefore, the next objective in graphene growth is to reduce the growth temperature using a more advanced CVD technology known as plasma-enhanced chemical vapor deposition (PECVD).

PECVD, as illustrated in Figure 4, utilizes energetic electrons, excited molecules or atoms, and free radicals in the plasma region as a thermal energy substitute. It also enhances the decomposition of reactant gases and promotes deposition of atoms onto the substrate surfaces while operating a CVD process at lower thermal conditions of approximately 650 to 750 °C (Zheng 2017).

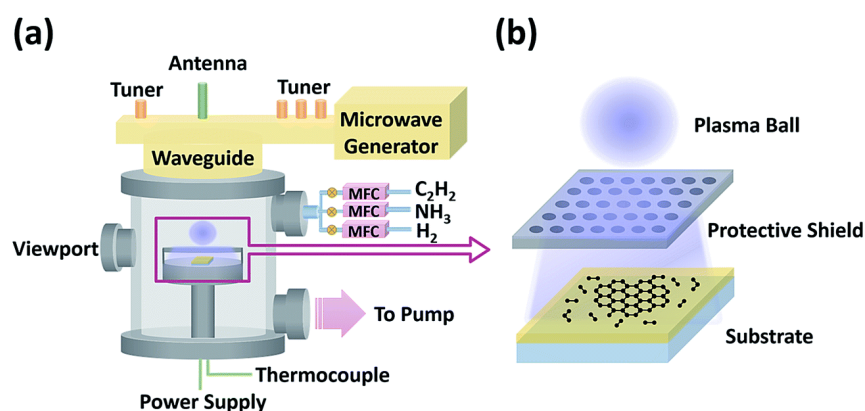


Figure 4. Schematic illustration of (a) PECVD system and (b) the zoom-in image of the graphene growth.

When highly energized plasma ions bombard the graphene film, it can lead to collateral damage and negatively impact the uniformity of film coverage, as well as degrade the film's quality and alter its electric properties (Jang 2015). Therefore, it is crucial to carefully control the dose and energy of plasma ions to minimize the potential damage and optimize the quality of the graphene film. In addition to the issue of ion bombardment, the byproducts generated during all CVD processes are predominantly toxic and volatile, which can pose health risks if inhaled. Therefore, a crucial goal in the current graphene research is to develop a more economical and safer method for producing high-quality graphene films with controlled thickness and uniform monolayer coverage. This requires exploring new techniques and refining existing methods to minimize potential hazards, reduce costs, and improve the overall quality of the graphene films produced.

1c. Approach & Methodology

[Author: FC; Editor: NW] While graphene synthesis has already been established, most if not all techniques require elevated temperatures which our semiconductor substrate, GaN, or other dielectrics $Al_{1-x}Ga_xN$ will not be able to tolerate. It is therefore important to grow graphene at the lowest temperature in order to preserve material integrity. We will be combining two aforementioned processes by coating GaN with a polymer through a dip coating process and then incorporating a PECVD method for graphene growth. However, prior to undergoing PECVD, polymer conversion to graphene will be carried out through a thermal annealing process in order to characterize the suitability of our polymer to graphitize.

When selecting a polymer, we hypothesized that if it had a high density of sp² carbons and conjugation (i.e delocalized electron network), conversion to graphene could potentially be more feasible at lower temperatures (Figure 5). This hypothesis comes from the fact that graphene, at its molecular level, is a conjugated electron-delocalized sp² carbon network. That being said, we chose polyimide (PI) as our polymer of interest that could then be compared to polymethyl methacrylate (PMMA) since PMMA has been shown to undergo graphitization at temperatures of about 1750C (Mattias Kruskopf *et al* 2016 *2D Mater*). Additional support for our polymer hypothesis came from a research paper which showed that PI could be transformed to graphene at lower temperatures via a PECVD process, albeit it also included the use of a catalyst (Lu, Leu, and Yeh 2021). Lastly, after consulting with chemists, ex-professors, and scientists in related fields, the decision to use PI resonated. If PMMA could be converted to graphene at elevated temperatures, it was our hypothesis

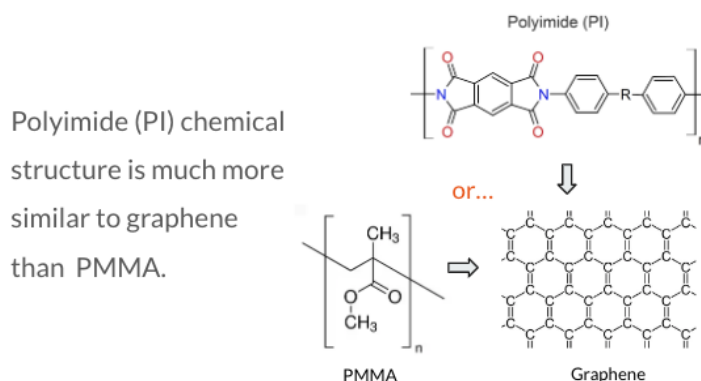


Figure 5. PI chemical structure compared with the chemical structure of PMMA and Graphene.

that a polymer with a more similar molecular structure to graphene could potentially be a better precursor.

Finding commercial polyimides posed challenges. Traditional chemical vendors (e.g. *Alfa Aesar* and *Sigma-Aldrich*) did not carry polyimides in unprocessed forms but rather supplied them as premade films. This problem was overcome by perusing LinkedIn for polyimide specialists and eventually contacting *Tetramer Technologies*, the molecular architects. We were able to connect with an employee and set up a couple of phone calls where we described the scope of our project and why we hypothesized that polyimides would work well. They agreed with our reasoning and proposed a couple of different polyimides for our project, which they eventually agreed to provide gratuitously.

For fabricating thin polymer films on GaN, we will be using an **Ossila** dip coater where our variables of focus are the withdrawal rate from the polymeric solution and viscosity. To stabilize the substrate during the dipping process, we have designed a substrate holder which was 3D printed and is illustrated in Figure 6.



Figure 6. A close-up view of the substrate holder working with a dip coater to coat the substrate.

The withdrawal rate determines the amount of material coated on our GaN substrate, allowing for flexibility in deposition. Another variable is the viscosity or polymer concentration of our solution, which determines the amount of polymer deposited on the GaN substrate after drying. Before conducting dip-coating on GaN, we will utilize a different semiconductor, SiO₂, due to its lower cost. This approach will provide us with a larger sample size for optimizing our dip coating parameters without incurring additional expenses.

In order to characterize graphene formation we will employ Raman spectroscopy as it is a standard nondestructive tool for the characterization of crystalline, nanocrystalline, and amorphous carbons, including graphene [11]. Raman spectroscopy is a technique for studying the vibrational, rotational and low frequency modes of vibration of molecules by illuminating the material of interest with a laser. Aside from being a nondestructive tool, graphene reference spectra from Raman spectroscopy already exist thus simplifying the characterization process.

For quantifying and characterizing the thickness of our films on the semiconductors via dip coating and then post thermal annealing, we will be using a measuring technique known as profilometry. Profilometry is a surface metrology technique used to measure and analyze a surface topography by using a stylus or a non-contact probe. The information gathered from the technique allows for a 3D

representation of the material probed. If we are successful in fabricating ultra-thin films, then using Atomic Force Microscopy (AFM) is in order. Without going into too much detail, it is the industry standard for characterizing films whose thicknesses are in the fractions of nanometers (graphene is 0.345nm), due to its very sensitive probing cantilever which interacts with a material's atomic forces.

Section II. Technical Executions & Outcomes

2a. Experimentation and Data

[Author: BT; Editor: FC, NW] *Substrate Preparation* - A three-step aqueous cleaning process is employed to remove any contaminants and impurities on the surface of our substrate. The substrates are consecutively immersed in acetone, isopropanol, and DI water for 10 minutes each, followed by drying on a hotplate at 70°C for 15 minutes. Just prior to dip coating the semiconducting substrates, they are ozone treated for 10 minutes in order to pacify the surface and enhance film adhesion from the dip coating process.

PMMA and PI Graphitization - To assess whether PI or PMMA is better for graphitization, these polymers were thermally annealed in either argon and hydrogen. Two 5x5mm GaN semiconductors were drop-casted with PI-A, another two with PI-B, and another two with PMMA resulting in six samples. These were then dried at 70°C for 30 minutes. Each sample was individually thermally annealed at 900°C for 1hr. After annealing, all samples treated with argon appear as if the film had vanished/vaporized. In hydrogen PI samples resulted in black films and PMMA appeared to have been vaporized. (*Note: PI-A was determined to be the better polymer for graphitization. See Results for explanation*).

PI Viscosity Optimization - As received PI-A solution has a viscosity of 100cP and is very concentrated. Volumetric dilutions of 50%, 75%, 80%, 87.5%, 92.5%, and 97.5% in N-methyl-2-pyrrolone (NMP) resulted in viscosities of 21.1 cP, 8.4 cP, 5.1 cP, 3.7 cP, 3.1 cP, and 2.5 cP, respectively.

Dip Coating Process - Dipping speeds, from 0.01 mm/s to 5 mm/s, a dipping speed of 3 mm/s were visually explored to best film depositions. The optimal withdrawal rates were determined to be 1 mm/s and 3 mm/s. Two rates were selected due to their better reproducibility visual profiles. A range of hanging times were also tested, varying from 0 to 10 minutes. Vertical hang time of 3 minutes was determined to be the optimal duration for achieving the best visual uniformity. The dipping cycle has been set to 1, indicating that each substrate is only dipped once in the coating solution. The dwell time, or the time that the substrate is held in the solution, has been set to 8 seconds. After dipping, a hot plate is used to dry the film at a temperature of 50°C for 15 minutes.

Thermal Annealing - Samples were annealed at 900°C with flowing H₂ at 17 sccm for one hour. In order to get clearer peaks and further graphitization, later samples were annealed at 1000°C for the same time.

2b. Results and Analysis

[Author: NW; Editor: FC, BT] *PMMA and PI Raman Spectroscopy* - Figure 7 shows that for both PI-A and PMMA (labeled PolymerA and PolymerB, respectively) annealed in argon largely etched away our polymers.

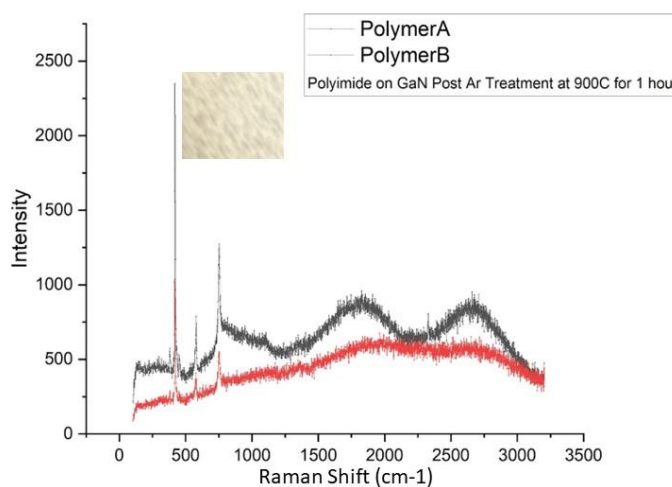


Figure 7. Raman spectroscopy for polyimide (PI) with polymer A & B post Ar annealing at 900°C for 1 hour, with an optical image of polymer A showing bright color post annealing.

Annealed in hydrogen, PI's and PMMA Raman spectra are presented in Figure 8. Although both PI samples showed encouraging signals, we selected PI-A due to its better D/G peak ratio of 0.95, which suggests a more efficient graphitization process. Additionally, it should be noted that PI-B was so statically charged due to ionization from thermal energy that it flaked off GaN. With these results, we proceeded with PI-A for the dip coating process as well as selecting hydrogen as our gaseous environment.

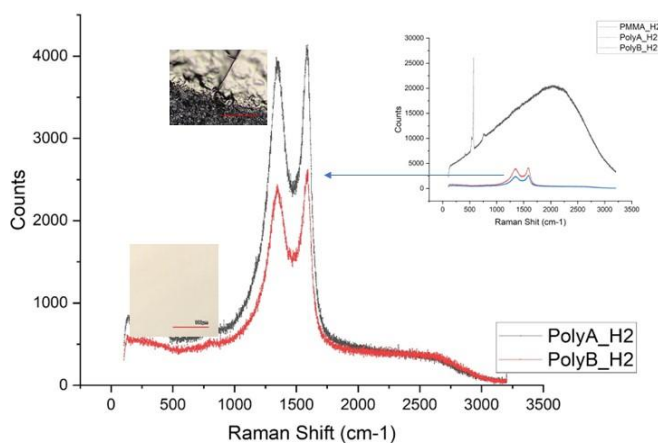


Figure 8. Raman spectroscopy for PI-A,B & PMMA post H₂ annealing at 900°C for 1 hour, with an optical image of PI-A showing blackened color.

Dip Coating Parameters - Numerous dipping trials have been performed to find the optimal dipping parameters such as dipping speed, vertical hanging time, dipping cycles and immersion time. A visual of the controlling process is illustrated in Table 1 below.

Viscosity (cP)	Dipping Speed (mm/s)	Withdraw Speed (mm/s)	Cycle	Dwell Time (s)	Hot Plate Temp (°C)	Hot Plate Time (s)	Vertical Hanging Time (min)
3.7	3	Initial: 1 Final: 3	1	8	50	15	3
3.7	3	Initial: 1 Final: 3	1	8	50	15	3
3.7	3	Initial: 1 Final: 3	1	8	50	15	3
2.5	3	Initial:1 Final: 3	1	8	50	15	5
2.5	3	Initial:1 Final: 3	1	2	50	15	5
2.5	3	Initial: 3 Final: 3	1	0	50	15	0

Table 1. A representation of the controlled process on the optimization of dipping parameters.

Optical Microscope - A dipping speed of 3 mm/s was determined to produce the most uniform film by observing the surface under the optical microscope, as demonstrated in Figure 9. Films produced at other speeds, shown in Figure 10, exhibited visible ripples when observed under a microscope.



Figure 9. Polymeric film pulling by 3 mm/s

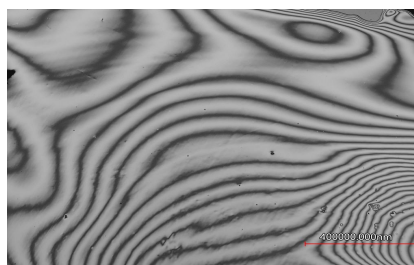


Figure 10. Polymeric film pulling by 0.09 mm/s

This demonstrates that, If the dipping speed is too slow, there is a risk that the fluid may start to dry out before it has spread uniformly over the entire surface of the substrate, which can lead to the formation of ripples and other surface defects.

The withdrawal rate for removing the substrate from the coating solution has also been tested and adjusted to be an initial withdrawal rate of 1 mm/s and a final withdrawal rate of 3 mm/s. It is

important to note that the withdrawal rate cannot be too high, as this can cause the coating solution to be rapidly pulled away from the substrate, leading to uneven deposition and the formation of ripples as well. Moreover, to achieve optimal film uniformity, controlling the vertical hanging time of the dipped substrate is crucial. This allows excess fluid to drain off from the substrate before it is dried or cured. In this study, the vertical hanging time was set at 3 minutes producing a surface image shown in Figure 11, as excessive hanging time can cause the coating solution to settle and form sediment at the lowest edge of the substrate due to gravity acting on the fluid, which can interfere with the uniformity of the thin film as demonstrated in Figure 12.

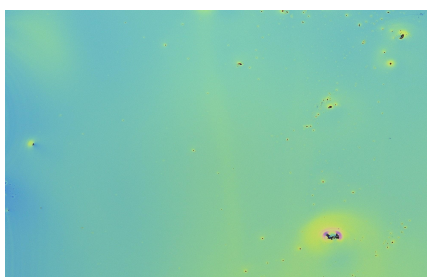


Figure 11. Polymeric film hanging for 3 min

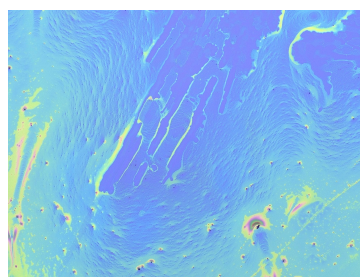


Figure 12. Polymeric film hanging for 7 min

Using the selected promising polymer and refined coating parameters, a series of dip-coating and annealing trials were conducted for multiple concentrations. The more concentrated samples with a viscosity larger than 3.7 cP were found to be unsuitable, as they failed to fully graphitize and resulted in the formation of an amorphous carbon structure after annealing. Finally, a promising result was obtained using the 3.7 cP polymeric solution, which exhibited a G peak at 1589 cm^{-1} and an intensity D/G peak ratio of approximately 0.773 at the thin edge of the film shown in Figure 13. The intensity D/G peak ratio is desired to be as small as possible (close to 0) to achieve a less disorganized carbon network with nearly no defects. These results suggest a successful graphitization process and the goal-approaching formation of graphite films.

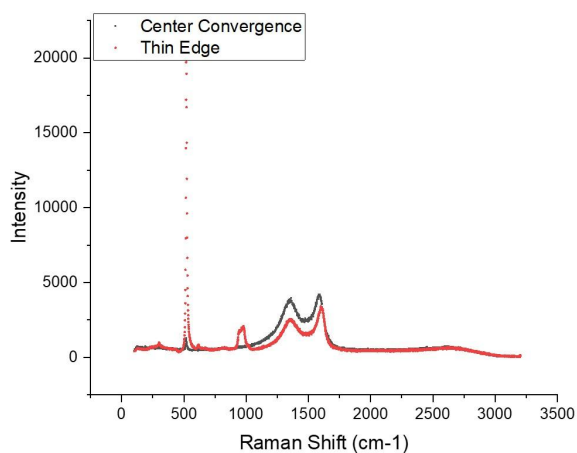


Figure 13. Raman spectroscopy for PI-A solution with 3.7cp of viscosity, post H₂ annealing at 900°C for 1 h

Figure 14 is a compilation of Raman spectroscopy on the graphite film annealed under different conditions. Though a graphene signal peak didn't show, an increasing trend of signal to noise ratio was

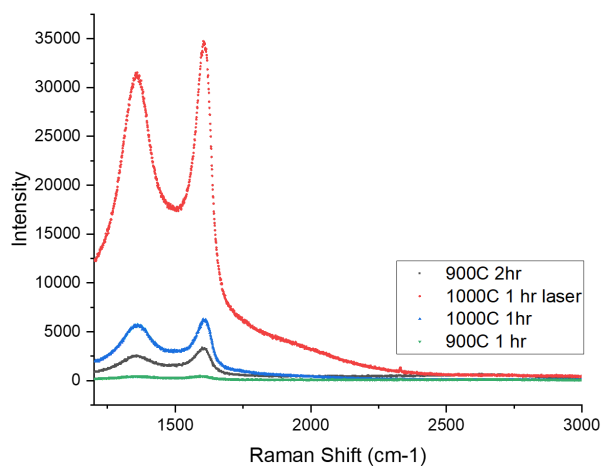


Figure 14. A compilation of Raman Spectroscopy on different annealing conditions. The strongest peak appears when multiple laser sweeps were performed with the aim to introduce laser-converted graphene. achieved by obtaining more uniform polymer films pre annealing. And the red curve was acquitted from 6 consecutive sweeps from Raman, from which we hoped to achieve a graphene conversion using laser energy.

VI. Conclusion

[Author: BT; Editor: FC, NW] By using a combination of a dip coating and a thermal annealing technique, graphite production from a polyimide was demonstrated, albeit having extremely large thickness of thousands of nanometers and of uncertain uniformity. Our optimal parameters for dip coating included a withdrawal rate of 3 mm/s, hang-drying for 3 minutes, and a solution viscosity of 3.7cP. Although we were unable to quantify reliable film thickness due to erroneous measurements observed during profilometry, we utilized Raman spectroscopy as an indirect measurement of our materials thickness. Given that we were never able to show a 2D band in our spectra indicative of sub nanometer thick films, we conclude that our polymer deposition process either needs further refinement or is not suitable for very thin film applications.

Dip-coating, on its own, has its limitations for producing very thin films with reproducibility. Such limitations may perhaps be overcome by coupling the technique with an added spin-coating procedure that can spread and thin out the film or other processes that can generate ultra fine, naked to the eye, films. In fact, for industrial thin film applications, ultrasonic spray coating is the standard deposition technique as the technique implies, ultrasonic waves are used at the spray nozzle to atomize the spray liquid into tiny droplets. Tiny droplets could also lead to potential film uniformity issues and perhaps then adding a spin coating step could be a solution to this potential problem.

Polyimides were superior for graphitization than PMMA perhaps due to their higher density of sp² carbons along with higher aromatic and conjugation content. It is important to note that exploring the graphitization potential for other polymers such as polyacrylonitrile (PAN) is also advisable (Zhang et al. - ACS Sustainable Chemistry & Engineering - 2022).

It was of great excitement to observe a black film formation on our drop-casting experiments with PI's in hydrogen given graphite (pencil lead) is black-grayish. Furthermore, the statically charged PI-B

was an interesting observation which implies an excess of electrons in the material (perhaps this could be used in a different application).

The refinement of graphene production is an ongoing process, with the aim of reducing contamination, lowering thermal costs, and achieving higher quality graphene films. The integration of graphene with Group III-N semiconductors has been a significant milestone in advancing the technology of hot electron transistors. Furthermore, the possibility of adapting this growth method for other electronic applications in a cost-effective manner is an attractive area of research. For example, there is potential for using graphene films to defog windshields, which could have significant practical benefits in automotive applications. Overall, the advanced method of graphene growth has the potential to revolutionize a range of industries and applications. The continuing research efforts in the field of graphene production and integration will continue to drive innovation and enable new possibilities in electronic and mechanical engineering.

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Academic Integrity

(1) NW - Neil Wang; BT - Bowei Tang; FC - Fernando Campos

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