

# An analysis of Graphene Synthesis

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**ABSTRACT:** Due to its remarkable physical, mechanical, and thermal characteristics, graphene has gained a lot of attention and interest in the scientific community. Graphene is a carbon allotrope with planar sheets of  $sp^2$  linked carbon atoms tightly packed in a honeycomb crystal structure that are one atom thick. Many techniques for synthesizing graphene have emerged in recent years, and we think it is essential to compile a list of the most noteworthy ones. This article focuses on graphene synthesis techniques in an effort to review and record progress in graphene synthesis research as well as future possibilities. Graphene, a one-layer thick substance, has isolated physio-chemical properties ranging from mechanical behaviour to electrical and even thermal dynamism. As a result, the world regards graphene as a miraculous substance. In this review article, several significant synthesis and future intelligent uses of semiconducting graphene are emphasized in light of recent advances. The emphasis is on the manufacturing processes, which include exfoliation techniques, Chemical Vapour Deposition on various metals, and various methods of graphene oxide reduction. Graphene is used in a variety of technologies, including solar cells, supercapacitors, thermoelectric devices, fuel cells, and even bio applications.

**KEYWORDS:** Conductivity, Exfoliation, Graphene, Graphite, Pyrolysis.

## 1. INTRODUCTION

Graphene is the most basic form of carbon and unquestionably the thinnest substance yet created. It contains planar sheets of  $sp^2$  bonded carbon atoms that are one atom thick and tightly packed in a honeycomb crystal lattice. Understanding graphene as a few layers of graphite is helpful. The remarkable characteristics of honeycomb carbon are not unexpected in this context. The layered morphology and modest dispersion forces between neighboring sheets were used to create marking tools in the middle ages, similar to how pencils are used now. Graphite has recently been utilized as a dry lubricant instead of the more expensive hexagonal boron nitride and molybdenum disulphide. Graphite is also utilized as electrodes because to its strong in-plane electrical conductivity (104/cm) and excellent thermal conductivity (3000 W/mK) in industrial blast furnaces [1].

Boehm et al. were the first to coin the word "graphene" to describe single-layer carbon foils. Graphite and the suffix '-ene' were used to create it. Graphite flakes are a stack of graphene layers with an interplanar spacing of 3.5Å and a C-C bond length of 0.142Å. Graphene is a single atomic plane of graphite that is sufficiently separated from its surroundings to be called free-standing", according to Wikipedia. Graphene seems to be a promising material for creating functional nanocomposites with outstanding characteristics. The fact that its predecessor (graphite) is widely available just adds to the allure. Defect-free graphene has exceptional physical properties, including high intrinsic mobility and ballistic transport, high thermal conductivity and Young's modulus, almost 98 percent optical transmittance, and a large specific surface area. We shall not discuss the characteristics of graphene since the characterisation of graphene and its derivatives has received a lot of attention. This review article focuses on the history of graphene production as well as current advancements [2].

### 1.1 Synthesis of graphene:

Graphene has been in the spotlight for some time, and many academics have been working on its synthesis. As a result, various techniques for the exfoliation of graphite into graphene have been described. The top-down approach and the bottom-up method are the two major categories. Graphite is essentially a stack of graphene layers, and van der Waals forces must be overcome in order to divide these layers into individual graphene sheets. The top-down strategy is the name for this method. Surface flaws that arise during sheet separation and the separated sheets re-agglomerating subsequently are two difficulties connected with this technique. Top-down methods, in general, provide poor yields and are time-consuming procedures. While the top-down method focuses on splitting a stack of graphene precursor (graphite) into atomic layers, the bottom-up approach utilizes carbon molecules as building blocks, which are usually acquired from other sources.

Although the bottom-up method is not suitable for the manufacture of large-surface-area graphene sheets, it does allow for the large-scale production of graphene nano-ribbons and graphene dots (so-called nanoflakes). After the discovery of graphene, the most frequent technique used to manufacture high-quality, defect-free graphene was micromechanical cleavage of graphite. This process is also known as "scotch tape," "drawing,"

or "peel off." Chemical vapour deposition (CVD) became a feasible option after a while. However, it was quickly discovered that these methods had a poor manufacturing yield, and that they were time intensive, especially in the case of micromechanical cleavage, preventing effective and complete utilization of these materials. Despite the fact that researchers have fine-tuned the method to produce high-quality crystallites up to 100 m in size, it is still difficult to separate in intercalated graphite compounds (so-called GIC) [3].

### *1.2 Top-down approach:*

#### *1.2.1 Mechanical exfoliation:*

The mechanical or micromechanical exfoliation technique has been a watershed moment in graphene's history. It is still the most important synthesis method for producing high-quality graphene for research purposes, as shown by TEM and AFM examination of graphene films ranging in size from 5 to 10 m. However, the inconsistent thickness of the films produced by this technique, as well as the expensive cost of manufacturing owing to the poor yield, make this technology unsuitable for mass production. These techniques are an excellent example of a top-down strategy, in which a graphene precursor (graphite, GO, etc.) is dismantled layer by layer, resulting in graphene sheets. Graphene was created through micro-mechanical alleviation of graphite by a group from Manchester University that included two Nobel Laureates, Geim and Novoselov.

They utilized a technique known as "scotch tape" or "peel off" to repeatedly break graphite crystals into thinner and thinner bits. After dissolving the tape carrying optically transparent flakes in acetone, the flakes comprising both multi-layer and monolayer graphene were sedimented on a silicon wafer to be examined under a microscope. Researchers subsequently modified this technique to skip the period when graphene floated in a liquid. As a result of these developments, graphene flakes bigger than 1 mm became visible to the human eye. The present research surge is due to the ability to isolate graphene. Free-standing atomic planes were previously believed to be impossible to find. Scientists assumed that such structures would be unstable if scaled down to nanometers, and that they would scroll and bend. Some researchers think that inherent microscopic roughening on a 1 nm scale may have a role in the stability of 2D crystals [4].

#### *1.2.2 Graphite intercalation:*

Intercalation may also be used to reduce graphite to graphene. To make graphite intercalation compounds, different chemical species may be inserted between graphite interlayer gaps (GIC). The distance between graphite layers increases due to the presence of these intercalants. The increasing interlayer distance alters electronic interaction between graphene layers, which modifies the characteristics of graphene. Distinct intercalants may cause GIC to have a different set of characteristics that may be used in applications that need electrical, thermal, or magnetic performance [5].

#### *1.2.3 Nanotube slicing:*

Carbon nanotubes have also been used as a graphene precursor. This method can produce graphene nanoribbons with a diameter of a few microns. These methods produce high yields of high-quality graphene. Carbon nanotubes may theoretically be broken open to create 2D graphenesheets. Several techniques have been devised, including plasma etching of partially implanted nanotubes in polymer film and putting carbon nanotubes in a solution comprising potassium permanganate and sulfuric acid. Nanotube slicing can produce high-quality graphene. Chen et al. created a nanotube slicing technique that can be used in industry. Under a constant load of 0.06 N/cm<sup>2</sup>, carbon nanotubes were abraded between ground-glass surfaces, causing friction to slice them into graphene sheets. This method produces graphene that can be utilized in FETs, interconnects, NEMs, and composites [6]. Given the novelty of the precursor (CNT), however, this method may not be economically viable for commercial production.

#### *1.2.4 Pyrolysis method:*

Pyrolysis is a simple technique that may be scaled up for mass manufacturing. However, the yield is poor, and the final product contains contaminants. The graphene precursor: solid solvothermal product is obtained by heating a 1:1 molar ratio of sodium (2 g) and ethanol (5 mL) in a sealed reactor vessel at 220 °C for 72 hours. The residual product is then quickly pyrolyzed and rinsed with deionized water (100 mL). The suspended solid is then vacuum filtered and dried for 24 hours at 100°C in a vacuum oven. This solvo thermal reaction technique may provide a graphene yield of around 0.1 g per 1 mL of ethanol, with 0.5 g per reaction on average [7].

### 1.3 Bottom-up approach:

#### 1.3.1 Growth from metal-carbon melts:

A carbon source (e.g. graphite powder) is put in contact with a transition metal and heated to high temperatures, enough to melt the metal, in this simple technique. When carbon begins to dissolve in molten metal, the temperature may be reduced to reduce carbon solubility, resulting in excessive carbon precipitate. The precipitate may be skimmed off to yield various carbon forms, such as single layers of graphene, few layers of graphene (FLG), and thick graphite (SLG). Nickel seems to be the best candidate for this procedure since it is not Ramanactive. Ruthenium and iridium are also known to be useful, although the grade of graphene produced is inferior to that of nickel and copper. Several other variations of this method have been described.

However, graphene development on nickel films via CVD seems to be the most popular technique. Under inert conditions, nickel film was heated to 900 to 1000 °C (Ar gas used). Along with Ar, methane gas is injected into the system. This allows the methane gas's carbon to be absorbed by the nickelfilm. Later, the methane supply is cut off, and the solution is allowed to cool (under Ar flow), allowing the carbon to diffuse off of the nickel surface and form graphene sheets. This technique, like the nickel-based method, utilizes a Cu substrate and methane gas to deposit carbon on the surface at high temperatures and low pressure. However, air pressure causes multilayergraphene to form on both copper and nickel. This technique may be used to produce huge graphene films. Ethane and propane, in contrast to methane gas, form bilayer coatings; this may be owing to their molecular structure having a greater proportion of carbon.

#### 1.3.2 Epitaxial growth on silicon carbide(SiC):

Because of the thin graphene films (>50 m) that can be produced epitaxially on SiC substrate, this technique is suitable for usage in transistors and circuits. Graphene is produced in this method by heating silicon carbide (SiC) to 1100 °C. This technique yields epitaxial graphene, however the size of graphene flakes is determined by the size of SiC wafers. According to study, the thickness, mobility, and carrier density of graphene produced in this environment are influenced by the surface of SiC. In contrast to exfoliated graphene, graphene produced via this procedure has mild anti-localization. SiC-epitaxial graphene, on the other hand, exhibits extremely large, temperature independent mobility, similar to graphene obtained by drawing or peeling off, but not as high as exfoliated graphene. Graphene produced using this technique exhibits massless Dirac fermions even without transfer. Weak van der Waals forces responsible for multilayer cohesion in multi-layered epitaxial graphene do not always affect the electrical properties of individual sheets within a stack. The symmetry of interlayer interactions is linked to this effect. In other instances, such as bulk graphite, this process does not occur, and electrical characteristics are impacted. Cutoff frequencies of 100 GHz may be achieved on a 2-inch SiC wafer. Because the SiC substrate is costly and the yield is poor, this technique produces high-quality graphene at a high cost. As a result, this technique is unsuitable for industrial production.

#### 1.3.3 Dry ice method:

Graphene can be made using the dry ice method by burning 3g of Mg ribbon in a dry ice bowl and covering it with another dry ice (solid CO<sub>2</sub>) slab. Mg must be fully burned in CO<sub>2</sub> before stirring the residue overnight in 100 mL of 1 M HCl. Mg and MgO are both water soluble. As a result, the mixture is filtered, and the residue is washed with deionized water until the pH level is balanced. The residue must be dried overnight under vacuum at 100 °C to remove the water content, yielding a graphene yield of 680 mg (92 percent)

#### 1.3.4 Deposition:

Many techniques for deposition-based graphene production have been described, primarily because to its practicality and scalability. Thin graphene films (675.5 cm) are produced using chemical vapor deposition (CVD) methods. These films are ideal for usage in applications such as touch screens, smart windows, solar cells, flexible LCD, and OLED due to their enormous size. CVD methods allow for the production of high-quality, large-area graphene films. The majority of CVD techniques are scalable, but they are not economically viable. Nanotube-reinforced graphene can be made using spin coating. By spin coating and annealing dodecyl-functionalized single wall carbon nanotubes (DF-SWCNT), stronger, more flexible, and conductive graphene is produced. The reported method used HIP co-produced SWCNT.

Liang et al. described the preparation of DF-SWCNT and trichloromethane solutions. Spin-coating was used to distribute a solution comprising DF-SWCNT and trichloromethane over a pre-treated Cu foil, which was subsequently annealed for 15 minutes at 1080 °C under H<sub>2</sub> and Ar flow, while maintaining the chamber pressure constant at 933.25 Pa. The samples were reportedly removed and then allowed to cool to ambient temperature. In contrast to CVD, supersonic spray provides a straightforward and cheap way to deposit



graphene films on a range of surfaces. This method improves graphene rather than synthesizing it. This technique can produce graphene with few or no imperfections, with ID/IG ratios as low as 0.22. This system employs kinetic spray deposition, which takes advantage of supersonic droplet acceleration through a converging and diverging de Laval nozzle. When exposed to a high gas (air) stream, R-Go atomizes [8].

## 2. LITERATURE REVIEW

Papageorgiou D et al. discussed Mechanical properties of graphene and graphene-based nanocomposites in which the current state of the graphene-family of materials' inherent mechanical characteristics, as well as the production and properties of bulk graphene-based nanocomposites, are extensively addressed in this study. The use of Raman spectroscopy in characterizing and studying the mechanical characteristics of graphene flakes and composites is shown. The mechanical characteristics of nanocomposites described in the literature are also examined, as well as the production methods for bulk graphene-based nanocomposites. We discovered a unique connection between the filler modulus, obtained from the law of mixtures, and the composite matrix after analysing hundreds of literature articles on graphene composites. This connection is shown to remain true over a broad variety of polymer matrices, indicating that the widely held belief that the filler modulus is independent of the matrices is wrong, which may explain graphene's apparent underperformance in certain settings. Even with extremely modest loadings, graphene may give substantial reinforcement to the final material, while the factors that have a major impact on the nanocomposite are extensively examined. Finally, the prospective uses and future prospects of graphene-based nanocomposite materials are addressed in terms of scaling up capabilities and probable advancements [1].

Novoselov K et al. discussed the rise of graphene in which they discussed how on the horizon of materials science and condensed-matter physics, graphene a fast rising star. Despite its short history, this purely two-dimensional material shows excellent crystal and electrical quality, and has already disclosed a cornucopia of novel physics and possible applications, which are briefly described below. Whereas commercial goods can confirm the validity of applications, graphene no longer needs any further evidence of its significance in terms of basic physics. Because of its unique electronic spectrum, graphene has spawned a new relativistic condensed-matter physics paradigm, in which quantum relativistic events, some of which are unobservable in high-energy physics, may now be replicated and verified in table-top experiments. Graphene, in general, is a theoretically new family of materials that are just one atom thick, and as such, it provides fresh insights into low-dimensional physics, which has never failed to amaze and continues to give fertile ground for applications [9].

Coleman K et al. discussed a manufacturing perspective on graphene dispersions in which they discussed how the use of graphene's remarkable physical characteristics often necessitates its dispersion in aqueous or organic environments. The concentration and stability of the dispersion must be suitable for the ultimate application. However, because to the strong contact between graphene sheets, it disperses poorly in all organic solvents with the exception of a few high boiling organic solvents. This article provides an overview of graphene dispersion applications as well as a discussion of dispersion methods, including the effects of shear, solvent, and chemical modification on graphene dispersion (including graphene oxide and reduced graphene oxide). Manufacturing and commercialization are addressed in relation to these methods [10].

## 3. DISCUSSION

A two-dimensional sheet of sp<sup>2</sup>-hybridized carbon is referred to as graphene. Its honeycomb network, which can be stacked to create 3D graphite, rolled to form 1D nanotubes, and wrapped to form 0D fullerenes, is the fundamental building block of other significant allotropes. Long-range  $\pi$ -conjugation in graphene produces remarkable thermal, mechanical, and electrical characteristics, which have long piqued the attention of numerous theoretical investigations and have lately piqued the curiosity of experimentalists. On the horizon of materials science and condensed-matter physics, graphene is a fast rising star. Despite its short history, this purely two-dimensional material shows excellent crystal and electrical quality, and has already disclosed a cornucopia of novel physics and possible applications, which are briefly described below. Whereas commercial goods can confirm the validity of applications, graphene no longer needs any further evidence of its significance in terms of basic physics. Because of its unique electronic spectrum, graphene has spawned a new relativistic condensed-matter physics paradigm, in which quantum relativistic events, some of which are unobservable in high-energy physics, may now be replicated and verified in table-top experiments. Graphene, in general, is a theoretically new family of materials that are just one atom thick, and as such, it provides fresh insights into low-dimensional physics, which has never failed to amaze and continues to give fertile ground for applications. This paper discusses several aspects related to graphene.

#### 4. CONCLUSION

Since its inception, graphene has garnered a great deal of public and scientific interest. This is mainly due to its unique characteristics and the high-performance composites potential it offers. Because many techniques are not suited for scaling up, up-scaling graphene production is a problem on par with synthesis of high-quality graphene. Top-down and bottom-up methods have been used by scientists to manufacture graphene. Bottom-up methods, such as CVD, provide great thickness and quality control but are not suitable for industrialisation. It can, however, be utilized to make graphene for high-end applications like touch screens, OLED displays, and solar panels. In contrast to the bottom-up method, the top-down method entails breaking down graphene precursors such as graphite, GO, and other materials into a few layers of graphene. To date, a variety of methods for achieving this objective have been proposed. Shearing is an example of a method that is simple and scalable. Graphene is being synthesized on a large scale, however the present techniques depend on batch production, which has its own set of issues, such as difficulty maintaining property consistency, which is critical for industrial scale production. We think that graphene is not a situation where one kind of graphene is appropriate for all uses, which is one of the reasons why graphene produced using various techniques is suitable for usage in different applications. We expect to see a system that offers not only a fair balance between scalability and quality, but the best of both, because new techniques for synthesizing graphene are becoming more intriguing. The purpose of this article is to give an introduction and comparative research of graphene synthesis technique.

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