

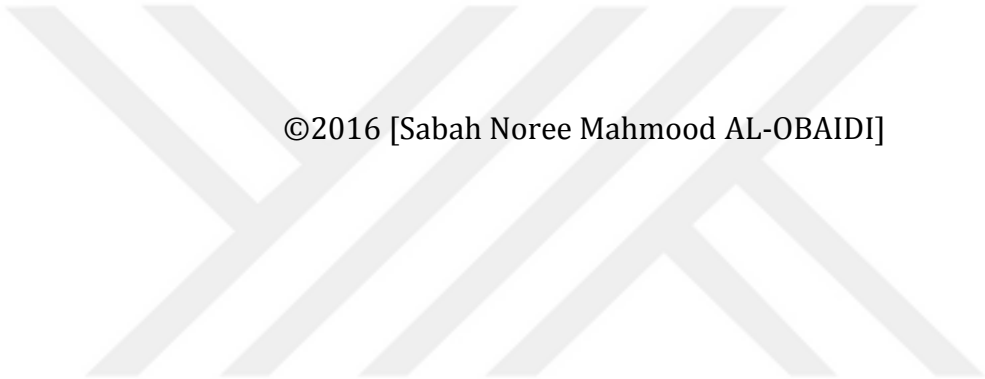
**T.C.
SÜLEYMAN DEMİREL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED
SCIENCES**

PLASMA INDUCTIVE DISCHARGE FOR GRAPHENE THIN-FILM

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**THE DEGREE OF MASTER OF SCIENCE
DEPARTMENT OF PHYSICS
ISPARTA - 2016**



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APPROVAL OF THE THESIS

" **Plasma inductive Discharge for Graphene Thin-Film** " submitted by **Sabah Noree Mahmood AL-OBAIDI** in partial fulfillment of the requirements for the **Degree of Master of Science in Department of Physics - High Energy and Plasma** , Graduate School of Natural and Applied Sciences, Süleyman Demirel University by.

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A handwritten signature in blue ink, consisting of a large, stylized 'S' followed by a smaller, more complex mark that appears to be '93' or similar, all written over the printed name.

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ABSTRACT

Master of Science

PLASMA INDUCTIVE DISCHARGE FOR GRAPHENE THIN-FILM

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Graphene sheets were produced by injecting ethanol molecules into radiofrequency plasma. The plasma enhanced chemical vapor deposition system (PECVD) was set up by using 40.68 MHz 250 W rf power source, a quartz tube (145 cm length and 2.5 cm radius) and the oven that partially covered the quartz tube. A copper sheet was placed inside the quartz tube for graphene deposition. Ethanol vapor was sent into the 1 mTorr argon environment and power was turned on for plasma. The oven temperatures were varied between 650-950 °C. The copper sheet surface were analyzed by XRD and SEM

Keywords: graphene, RF, Ar plasma, ethanol, H₂

2016, 84 pages

ÖZET

Yüksek Lisans

İNDÜKTİF DEŞARJ PLAZMASI KULLANILARAK İNCE FİLM GRAFEN SENTEZLENMESİ

Sabah Noree Mahmood AL-OBAIDI

Süleyman Demirel Üniversitesi

Fen Bilimleri Enstitüsü

Fizik Anabilim Dalı

Danışman: Prof. Dr. Lütü ÖKSÜZ

Radyofrekans plazma içine gönderilen ethanol molekülleriyle grafen tabakalar üretildi. Plazma yardımcı kimyasal buhar biriktirme sistemi, 40,68 MHz 250 W rf güç kaynağı, bir kuartz tüp(145 cm uzunluğunda ve 2,5 cm yarıçapında) ve kuartz tüpün bir kısmını saran fırın kullanılarak kuruldu. Bakır tabaka kuartz tüp içine grafen biriktirme için yerleştirildi. Ethanol buharı 1mTorr luk argon ortamına gönderildi ve plazma için güç uygulandı Fırın sıcaklığı 500-900 °C aralığında değiştirildi.Bakır tabaka yüzeyi XRD ve SEM ile analiz edildi.

Anahtar Kelimeler: grafen, RF, Ar plazma, etanol, H₂

2016, 84 sayfa

Acknowledgements

About one year ago I was search for name a master project and ended up in one off the plasma physics experiments.

I want to put this page lively moments of the conclusion of this study, and I am proud of them and seize the opportunity to thank those people who were necessary to do this work.

So i would like to thank everyone who has supported me during this master thesis project, a big thanks to my professor (Prof. Dr. Lutfi oksuz) my supervisor for letting me do this project by and for his advice which helped me on my project and for spending so much time on helping and supporting me during this thesis work. And I would like to thank everyone in the own group for great support. Especially for Dr. Ali Güleç' ,Ferhat BOZDUMAN and at end for my dear uncle Ramazan KILIÇ .Actually Without my friends I would have forgotten the world outside the lab Last but not least I would like to thank my parents and my friends in the lab for their support in all things. At the end, I would like to thank to Project Management Unit of Scientific Research Projects Department in Suleyman Demirel University for supporting my research under contract (4715-YL2-16).

Sabah Noree Mahmood AL-OBAIDI
ISPARTA, 2016

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SYMBOLS AND ABBREVIATIONS DIRECTORY

Ac	Alternating current
AFM	Atomic Force Microscope
Ar	Argon gass
CNT	Carbon Nano Tube
Cu	Copper
CVD	Chemical Vapor Deposition
Dc	Direct cuurent discharge
2D	Two-Dimensional
ev	Elektron volth
E beam	An intense burst of fast electrons from a small particle Accelerator, used to excite a pulsed gas laser in which the gaspressure Is too high to permit an electric discharge
E	Electron charge
$\vec{E}$	Electric field
ECR	Electron Cyclotron Resonance
$\vec{F}$	Electric Force
FeCl ₃	Iron(III) chloride
ICP	Inductive Cuople Plasma
I	Cuurent unite
IC	An integrated circuit is a thin chip consisting of at least two interconnected Semiconductor devices, mainly transistors, as well as passive components Like resistors.
IPA	Isopropyl alcohol
J	Unite of energy
Kg	Killo gram mass unite
LF	Low Frequency
m	mass
mTorr	Unit of pressure measurement. Millitorr (mTorr) or 0.001 Torr. ~133.3 Pa
NEMS	Nanoelectromechanical systems
n _i	Ion density
n _e	Electron density
PMMA	Polymethylmethacrylate (Acrylic)
PVD	Physical vapor deposition
PECVD	Chemical vapor deposition enhanced plasma
RPE-CVD	Rchemical vapor deposition enhanced plasma
RF	Radio frequency
Raman	Regional Atmospheric Measurement and Analysis Network
v	Volum
V	Volth unite
ρ	The electron density
φ	Electric potential
λ _{De}	Debay length
SWC	Single-walled carbon
W	The symbol watt is a derived unit of power in the International system of Units (SI)

OES	Optical Emission Spectrometry
SEM	Scanning Electron Microscope
T-CVD	Thermal chemical vapor deposition
TEM	Transmission electron microscopy
λ	wavelength of Laser
'εθənl'	Ethanol or simply alcohol



1. INTRODUCTION TO PLASMA PHYSICS

1.1. Definition

“Plasma “as a physics term often indicates or refers to as the fourth state of matter (Fridman & Kennedy, 2004). According to another definition, plasma is an ionized gas and it is the result of a series of transformations such as solid to liquid, liquid to gas through heating and ventually ionized and transferrormed into plasma (Goldston R.J., Goldston, H., & Rutherford, 1995) . Francis F.Chen points out an important consideration that any ionized gas cannot be called plasma as; there is always some small degree of ionization in any gas (Chen F. F., 1985). Francis F.Chen described plasma as a quasi-neutral gas of charged and neutral particles that exhibits collective behavior.

In light of such arguments we can label it as :- the fourth stat of matter. Its can be described as ionized state that contains equal amount of positive and negative ions ,is affected by electric and magnetic spheres or fields. It is result of a collective ionisation of atoms. It may be very hot or cold and it occurs naturally but also can be manmade through generation and stabilization in a laboratory (Fridman & Kennedy, 2004)

Plasma is a mixture gaseous of electrons and positive ions. It may be fully ionized as in the sun or partially as in fluorescent lamps. (Alexander, 2010) Finally, we can add Michel Lieberman’s definition that plasma is a set or a group of independent charged particles moving in random bearing or random direction that is on average electrically and magnetically neutral (Lieberman & Lichtenberg, 2005).

1.2. Plasma Forms [Classification and types]

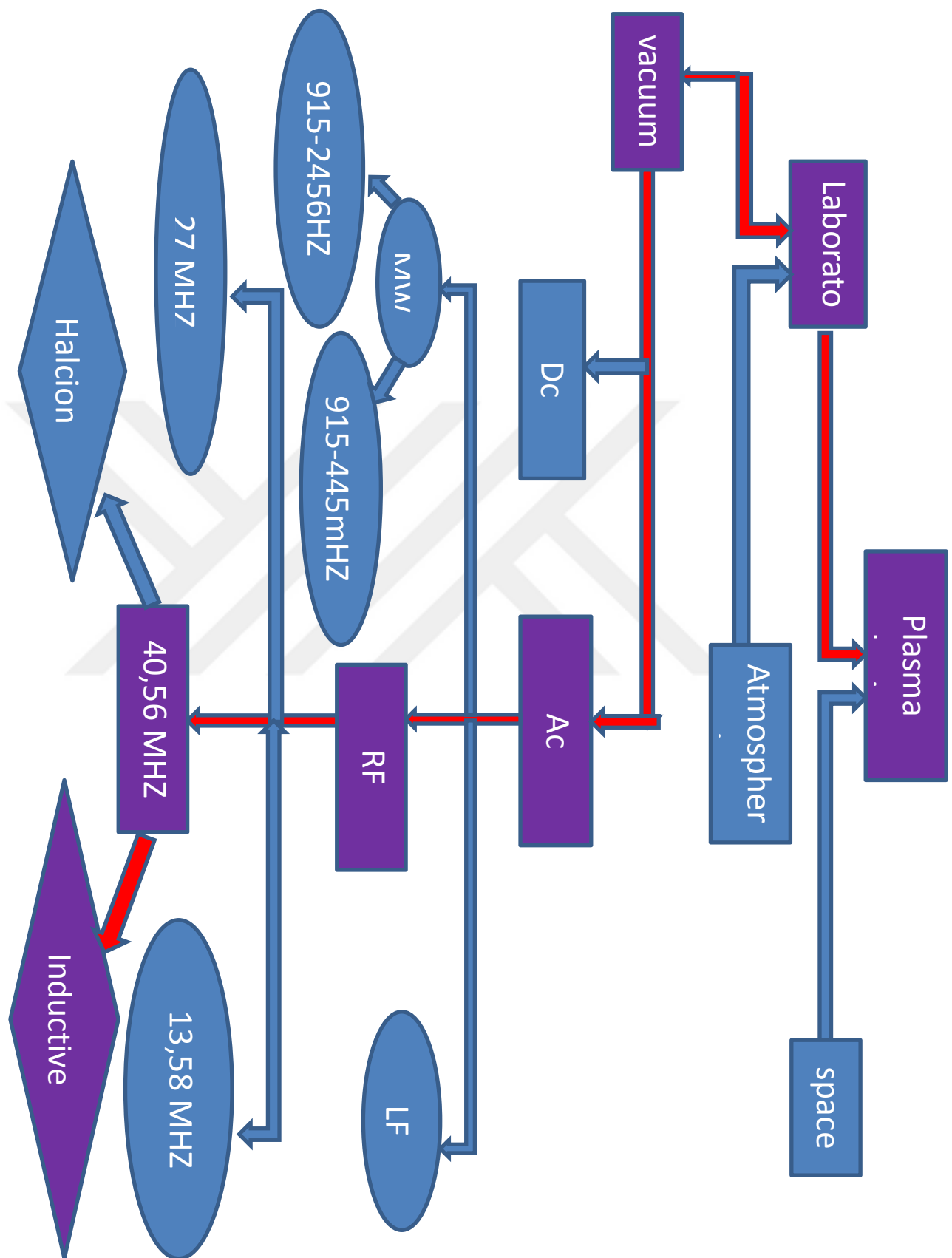


Figure 1.1. Plasma Classification and types

NOTE: - There maybe another division or classification which can complement this. It also is classified as following:-

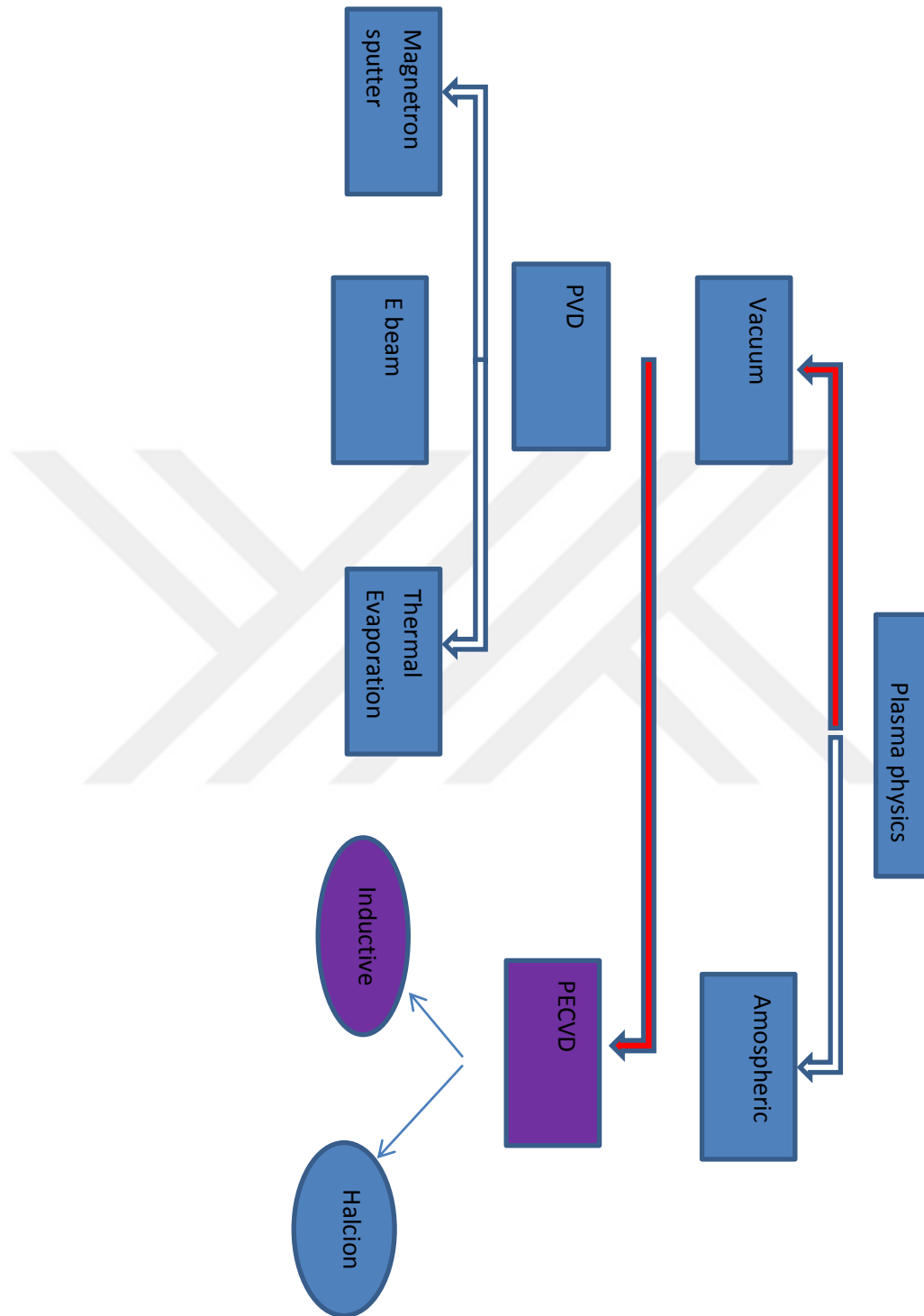


Figure 1.2. Another classification for plasma types

NOTE: - In both Figure, we can see Inductive and PECVD. So, we will talk first about the subject and later we will talk about plasma deposition (PECVD).

1.3. Parameters of Plasma

The physical portrayal of plasma requires specific time and length scales, and a base number of charged particles by unit volume. These parameters are associated with the reduction of the little changes of the congruity state of the plasma. The electric charge securing strategies in the plasma mass require different free charges to cross out the instabilities of the adjacent electric field. So, we have to have a base density of charged particles and this defines the asserted plasma parameter (López, 2011)

So we can sum up that there are three major parameters which describe plasma:-

- 1- The density of particle η (measured by m / v or Kg / m^3)
- 2- The temperature T of every part (each species) generally measured by ev
where $ev = 1.6 * 10^{-19} J = 11.605 K$
- 3- The steady state magnetic field B measured by Tesla or weber / $m^2 = 10^4$ Gauss

A host of subsidiary parameters (for example Debye length, plasma frequency, cyclotron frequency, thermal velocity, Larmor radius) can be derived from these three fundamental parameters. For partially-ionized plasma, the partial ionization and cross-sections of neutral are also important (Bellan P. M., 2008)

1.4. Basic Plasma Discharges

To create plasma, we must supply energy to gas to ionize it. This may be happen in many ways. One of the methods used for example thermally heating gas with a flame by compress the gas adiabatically or use supply energy by electromagnetic fields (SODERSTROM, 2008), most used technique in plasma technologies is use of an electric or electromagnetic field (SODERSTROM, 2008).

For this section or subject we will describe two basic types of plasma discharge. The first one is direct current (DC discharge) and the second one is radio frequency (RF discharge). So, we know that the bulk or the most of practical

applications for plasma processing use an Rf exciting plasma but Dc discharges remaining or it is simpler to analyses and to many of basic principles for the transition to Rf plasma (Lynn, 2011)

1.4.1. DC Discharges

Dc (direct current) discharge is usually called diode discharge since it is produced by applying a (Dc) voltage (Denis, 2009)

So the Dc discharge created by applying a Dc potential between an anode and a cathode inside of a chamber filled with a low pressure gas (Lynn, 2011). As shown in figure1.5. The anode and cathode are two conductive electrodes inserted into a gas at low pressure (Denis, 2009). When we apply voltage, it will create an electric field $\vec{E}$ which will act on the electrons contained in the gas as a result of ionization by a small amount of free electrons which is present in gas that occurred naturally by radioactivity or cosmic rays (Denis, 2009). So free electrons and ions that exist in gas from random thermal processes accelerated under the influences of electric field between the electrodes (Lynn, 2011) , this acceleration creates force $\vec{F}$ which can be calculated from equation

$$\vec{F} = e\vec{E} \quad (1. 1)$$

Where e is the electron charge when this electrons and ions gain energy it made collision with the gas atoms or molecules (Denis, 2009) , the collision mad or generate glow discharge and mad the sheath as shows in Figure 1.5.

The generation of glow discharge requires a minimum threshold voltage called breakdown voltage, this differ in voltage are illustrated in figure 24, the breakdown voltage it depends on:-

- 1- The gas
- 2- System pressure
- 3- Distance between the electrodes

Dc discharge can usually be shown as divided in to distinct regions (Söderström, 2008) as shown in figure 3.2.

We can illustrate the distinct regions of Dc discharge. So, close to the cathode there is a thin luminous layer is usually observed the cathode glows due to the atoms excited by electrons or ions which accelerated from the plasma toward cathode close this region the dark space between the cathode called glow(Aston dark space) as seen in Figure1.5 and 1.6. Moving towards the anode after the cathode glow, we can see a dark region called cathode dark space. It is often referred to as the cathode sheath, directly we can see the negative glow, after the negative glow another dark space follows which is called the Faraday dark space. The positive column following the faraday dark space which has a small electric field just large enough to maintain ionization. Finally, close to the anode we may see the anode glow followed by an anode dark space which is as opposed to the cathode dark space that has a negative space charge (Söderström, 2008) as we can seen in Figure 1.7, 1.8 and 1.9.

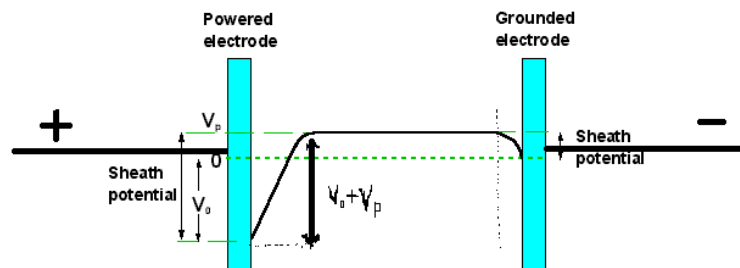


Figure 1.3: Voltage distribution in an example DC glow discharge process
(SODERSTROM, 2008)

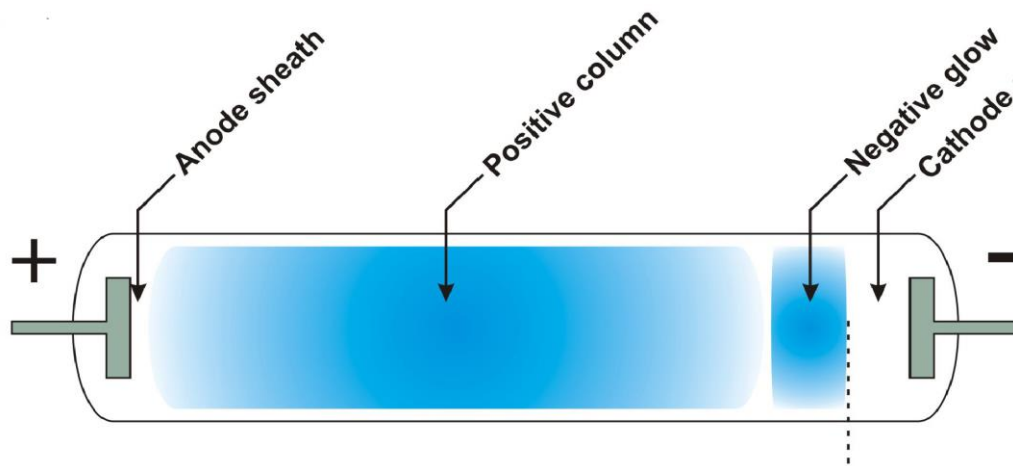


Figure 1.4: Regions of a DC glow discharge (SODERSTROM, 2008)

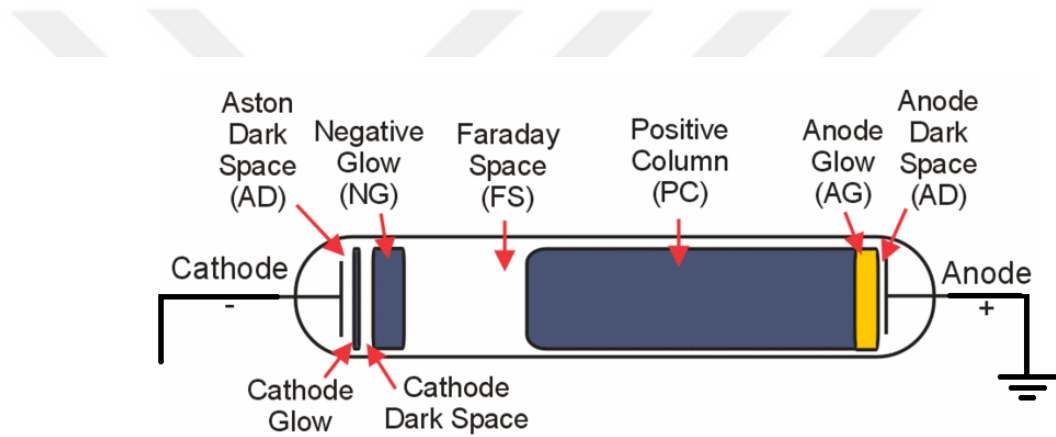
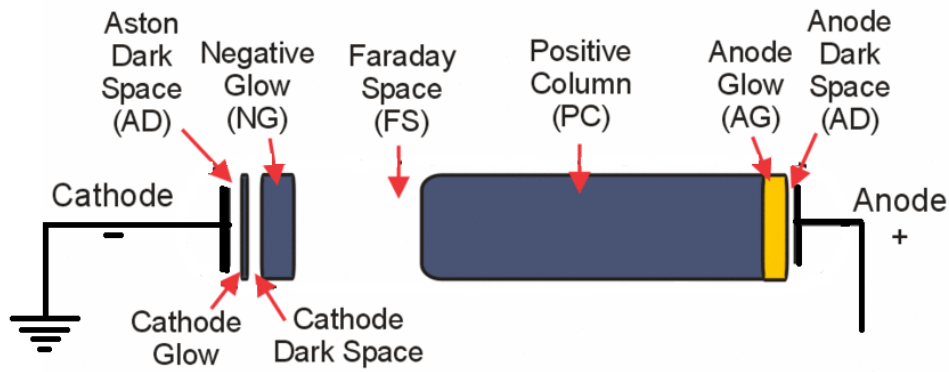


Figure 1.5: The structure of the DC discharge with dark and luminous regions, An electric glow discharge tube featuring its most important characteristics: (a) An anode and cathode at each end (b) Aston dark space. (c) Cathode glow (d) Cathode dark space (also called Crookes dark space, or Hittorf dark space). (e) Negative glow. (f) Faraday space (g) Positive column (h) Anode glow (i) Anode dark space (Söderström, 2008)



Dc discharge

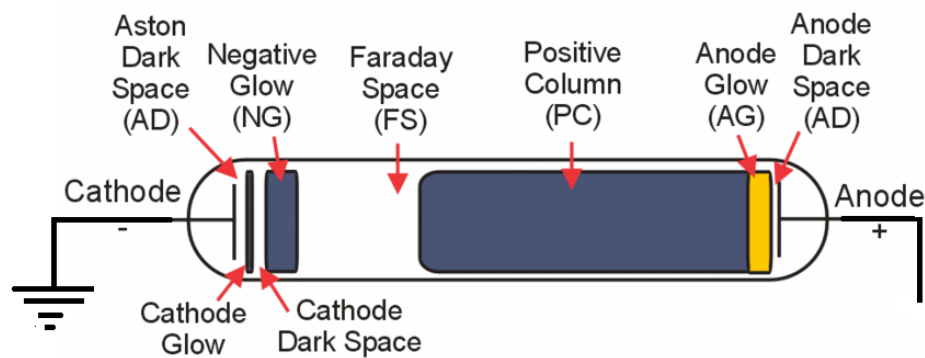


Figure1. 6: The DC discharge with dark and luminous regions .An electric glow discharge tube featuring its most important characteristics (Söderström, 2008)

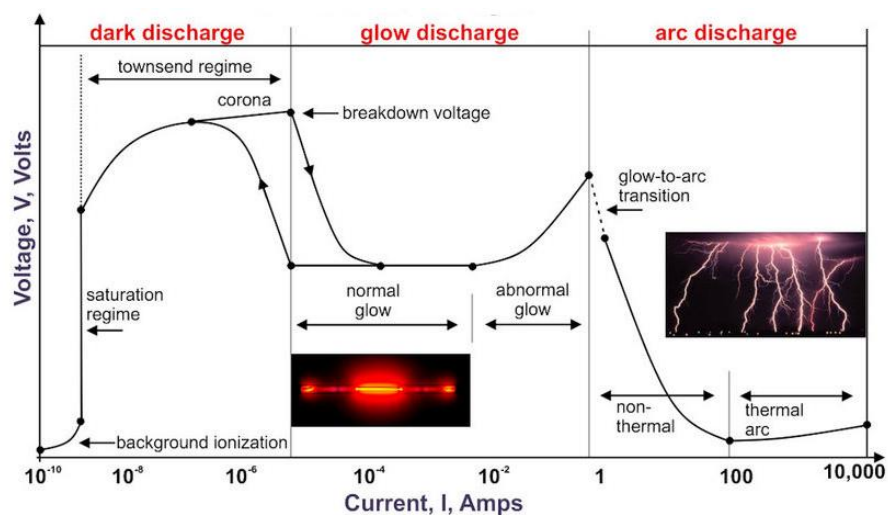


Figure 1.7: The different discharge regions for a DC discharge or electric glow discharge (Laboratory, 2015)

The above figure is a typical V/I plot of a glow discharge. The main characteristics of the discharge such as the breakdown voltage, the voltage current characteristic and the structure of the discharge depend on the geometry of the electrodes, the gas used, the pressure and the electrode material (Laboratory, 2015)

1.4.2. RF Discharges

Many of industrial applications of glow discharge processes are powered through using oscillating electric fields, with high frequency power supplies (Lynn, 2011). So, radio frequency (RF) discharges operate at frequencies of 1-100 MHz, which means that the generated electromagnetic waves have a wavelength of 300-3m generally much larger than the dimensions of the discharge plasmas. It is therefore possible to look at RF discharge as electrostatic when modelling it (SODERSTROM, 2008). By using RF discharges, the power will be coupled more efficiently to the electrons with RF, these discharges often have more efficient ionization (SODERSTROM, 2008). Radio frequency power is possible to interact with plasmas either capacitively, inductively or quasi-optically (Roth, 1995), yet the last one is not the subject of our study. So, we can say plasma can be generated by two types of RF system namely capacitive, and inductive. But the limitations of RF diodes and their magnetically enhanced variants have led to the development of new devices for generation of low-pressure, high-density plasma sources (Denis, 2009). Some examples are

- 1- ECR
- 2- Helicon
- 3- Helical resonator
- 4- Inductive

The first one, ECR (electron cyclotron resonance), is shown in figure 1.10

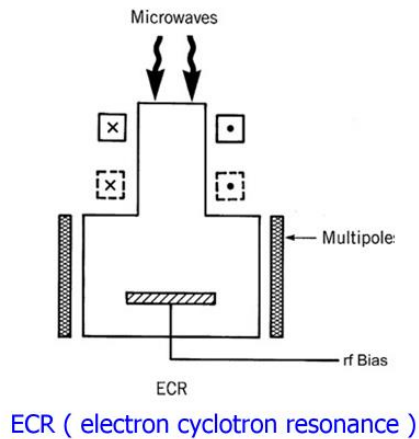


Figure 1.8. Illustrated ECR electron cyclotron resonance (Lieberman & Lichtenberg, 2005)

There are one or more electromagnet-coils surrounding the cylindrical source chamber that generates an axially varying DC magnetic field and microwave power is injected axially through a dielectric window into the source plasma (Lieberman & Lichtenberg, 2005). The plasma will appear like streams out of the source into the process chamber in which the wafer is located (Lieberman & Lichtenberg, 2005).

The second one is a helicon source as shown it in figure 1.11

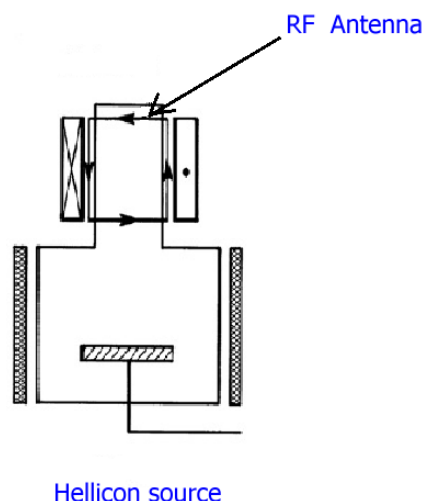


Figure 1.9. Illustrates the helicon source and RF antenna (Lieberman & Lichtenberg, 2005).

A weak (50 – 200 G) dc axial magnetic field together with an RF – driven antenna placed around the dielectric cylinder that forms the source chamber allows excitation of a helicon wave within the source plasma (Lieberman & Lichtenberg, 2005).

Now we will talk about the third one for the helical resonator source we can see it in figure1.12

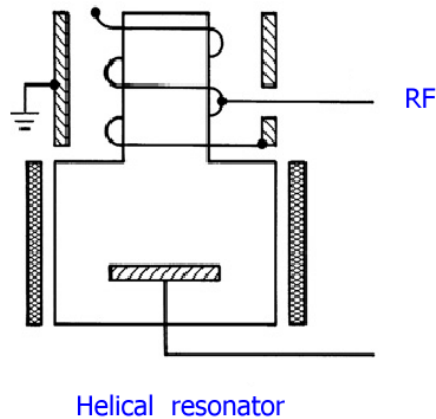
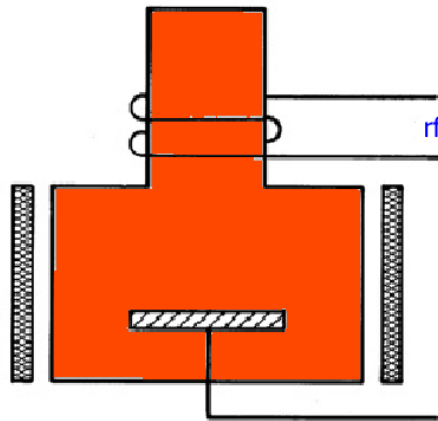


Figure 1.10. Illustrates the Helical resonator source and RF antenna (Lieberman & Lichtenberg, 2005)

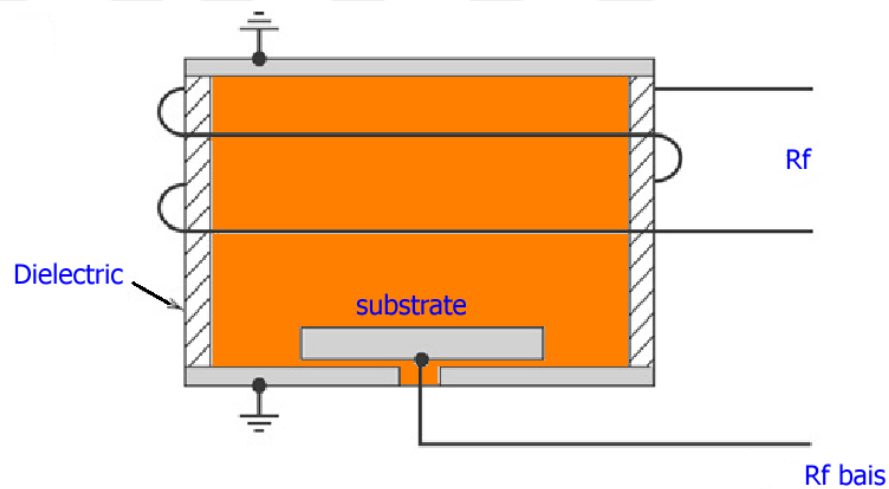
For Helical resonator source there is external helix and conducting cylinder surrounding the dielectric discharge chamber form a slow wave structure ,which supports an electromagnetic wave with phase velocity much less than the velocity of light (Lieberman & Lichtenberg, 2005).

Lastly, we can see an inductive in figure1.13 and 1.14



Inductive

Figure 1. 11. Illustration of the Inductive source and RF antenna (Lieberman & Lichtenberg, 2005).



Figur1. 12. Schematic of inductively driven sources in cylindrical (Lieberman & Lichtenberg, 2005).

Another type of RF plasma inductive discharges is nearly as old as invention of electric power. The first one who reported it was Hittorf in 1884 (Lieberman & Lichtenberg, 2005). An inductive contains an external coil; for example, wrapped around a dielectric vacuum tube carries an Rf current, which creates a varying magnetic field and in turn induces an electric field that accelerate the

electrons (SODERSTROM, 2008) we can see it in Figure1.14. But there is the question of why we use RF? We can answer in next part.

1.5. Why Do We Use RF?

There are many reasons or advantages for using RF discharge in plasma processes which includes

- 1-a (DC) bias cannot be used or applied to a semiconductor wafer in a processing chamber as some kind of layers of wafer may be made up of insulating materials¹.
- 2- Modulated by the RF frequencies, electrons can “collide“these, also rebounding into the discharge with a greater momentum.
- 3-The RF discharge does not have as many clearly defined regions as the DC discharges , RF discharges consist typically of three parts: the first one is the plasma bulk and the second and the third are two electrode sheaths (Lynn, 2011)
- 4- The massive ions respond only to the time- averaged electric fields. Oscillation of the electron cloud creates sheath regions near each electrode (Lieberman & Lichtenberg, 2005)
- 5- RF discharges are different from DC discharges since there are no dedicated anodes or cathodes, and no defined floating potential (Lynn, 2011)
- 6- Various magnetically enhanced RF discharges and triodes have also been developed to improve performance of the RF reactor (Lieberman & Lichtenberg, 2005)

¹ . ADC bias is useful when we use plasma processing applications to ensure that reactive species are attracted to the wafer surface in a directional manner [Shane Lynn, April 2011]

1.6. Classification of RF Discharges

Traditionally, classification of RF discharges depends on the mechanism of RF power coupling to plasma so they are either capacitive or inductive as we can see it in Fig1.15

In 1947, G. I. Babat was the first person who introduced the terms "E-discharges" and "H-discharges". He illustrated the difference between the plasma generations by magnetic or electric fields. The electromagnetic field maintains a discharge it causes by oscillating current exactly in antenna, so antenna can generate an electric field directly in inductively coupled plasma as we see in the Fig1.11.

There are two modes of operation known as "E-mode" and "H-discharges" (eines, Bochum 2004) .

We will use the latter in our experiment "H-mode" can be found in higher plasma densities, so H-mode deposits the RF power energy in plasma through accelerating the electrons by ring-shaped induction field inside the skin layer so the electron flow represents a ring-current which has opposite direction as the RF current in the catalyst coil. In this system the single winding or wrap for an air-core transformer includes multi-winding exciter coil and the skin layer of plasma (Piel, 2010)

Classification of RF Discharges

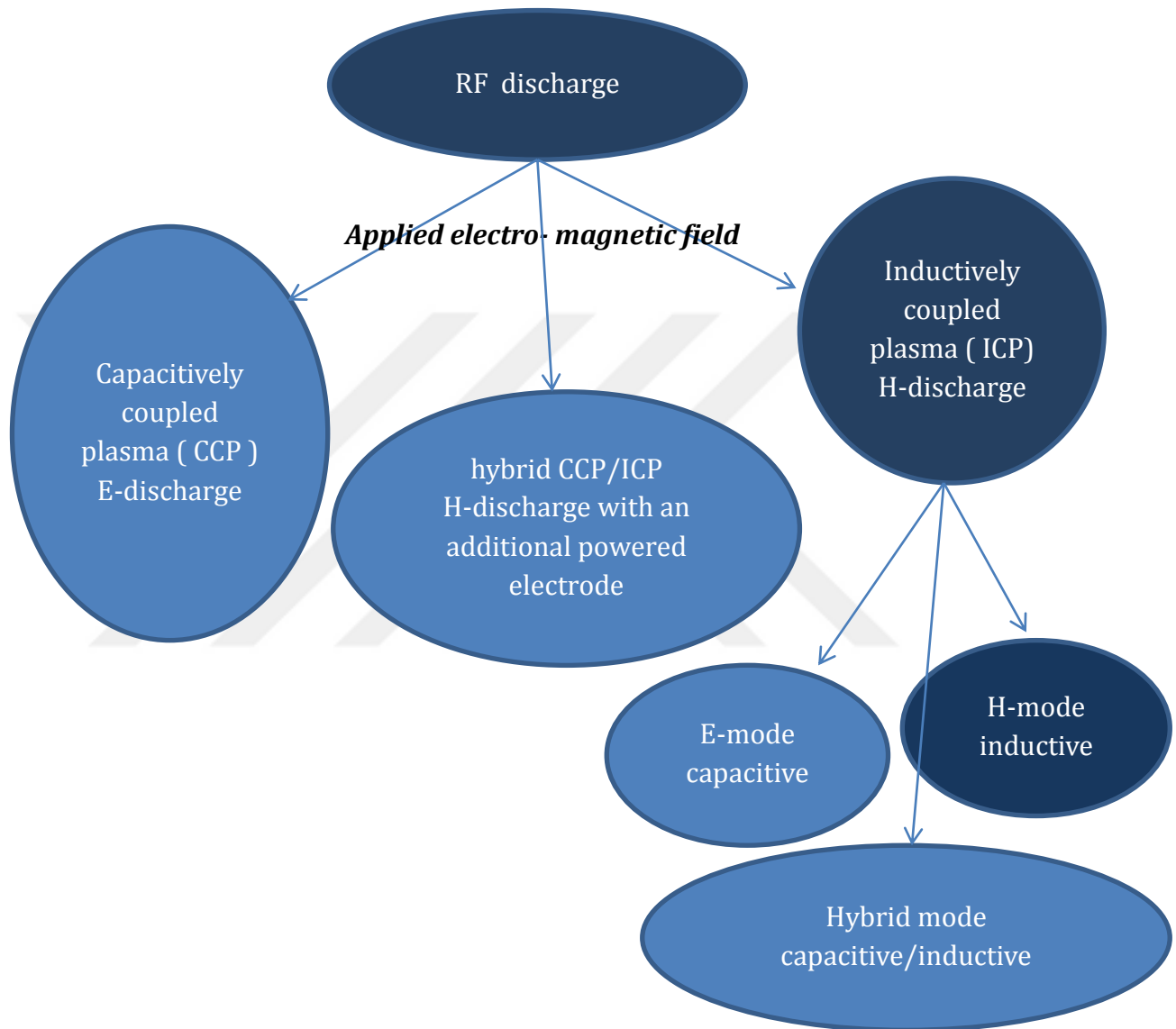


Figure 1. 13. Classifications of RF Discharges

1.7. Matching Networks

In order to achieve maximum power to be transferred from the power supply to the plasma, it is widespread practice to use a circuit which uses RF power source that includes a matching network circuit on the powered electrode supply circuit. So if we have RF power source, we cannot connect it directly to the plasma discharge because the power is not transferred efficiently in RF power source (Lynn, 2011)

From electrical science, we know that inductors and capacitors in any circuit have impedances with opposite signs. The inductors contain positive sign and the capacitors contains negative. So if we add an inductors which has impedance value to circuit, the capacitance and inductor will cancel each other and our match will be complete (Todorow, 2009)

There are another reason for using the matching network which is to protects the generator from any reflections that may be induced in the power circuitry (Lynn, 2011)

We can see the matching network in figure 1.16, 1.17 and 1.18

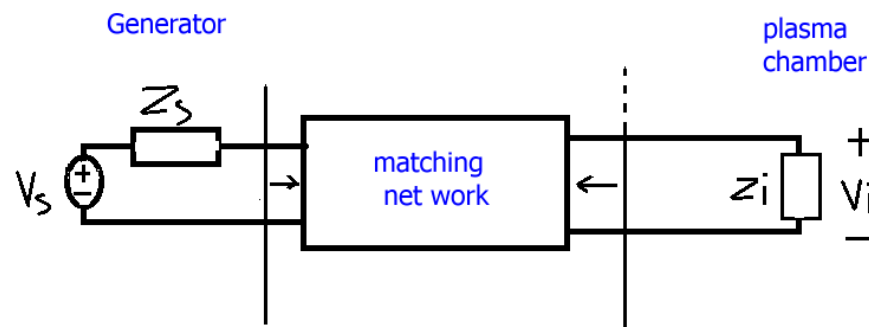


Figure 1.14. Matching network between the plasma and generator. The matching network is automatically controlled to ensure that maximum power is transferred between the generator and the discharge (Todorow, 2009).

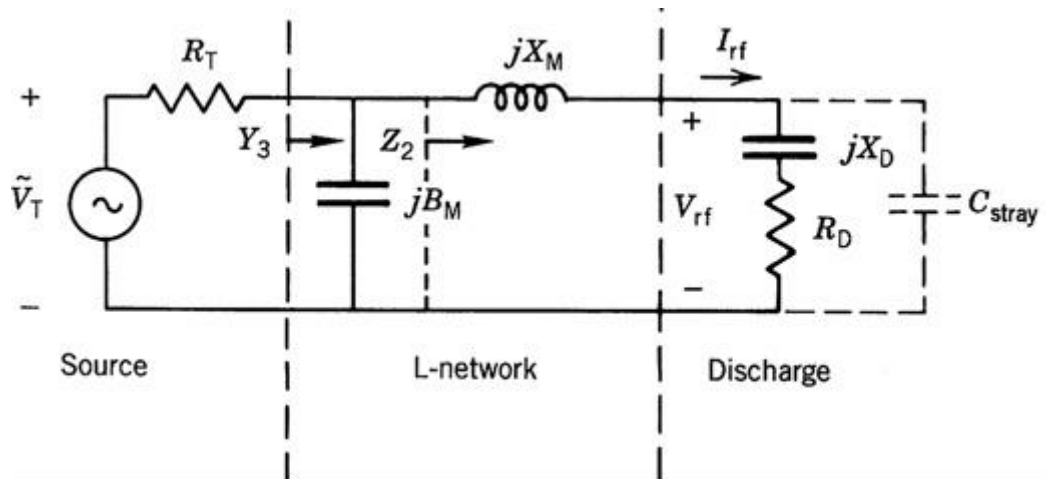


Figure1.15. Equivalent circuit for matching the rf power source to the discharge using an L-network. (Lynn, 2011)



Figure 1.16 Image for matching device. Impedance matching side view of unit (Andrew S., 2012)

2. PREVIOUS STUDIES

2.1. Previous Studies for Graphene Synthesis

In chapter one, we focused on plasma and its previous studies. In this chapter, we will focus on graphene. Firstly, we want to define what the term 'graphene' means. Graphene is a thin layer of pure carbon. It is single layer made tightly packed carbon atoms. These atoms are bond together in a hexagonal honeycomb lattice. Actually, graphene is classified as a semiconductor (Twain M. ., 2012). The beginnings of graphene discovery started at the second half of the nineteenth century. In 1859. Benjamin Collins Brodie discovered graphite oxide (Debye, peter & SCHERRER, 1916). The properties of graphite oxide which its structure was determined from single-crystal (Kohlschütter, 1919) .

The theory of graphene was first created by P.R.Wallace in 1947 as a starting point for understanding the properties of graphene. After that studies rolled one after an other (Boehm, 1962) (Meyer, 2007).

The first successful attempt of graphene synthesis was in October 2002, it was labeled as Nano-Scaled graphene plate. So if we study the attempts we will see the products of graphene.

Recently several important techniques have been developed to prepare nanostructured graphene (Tang, 2013).

The production methods are various. Some of the methods were chemical as chemical vapor deposition which denoted by CVD and some others were the physical as PVD (physical vapor deposition) and some were mixed between the two. We will explain the chemical methods first and then the physicals methods later. For tactical reasons we will only focus on a selection of studies as there are countless studies on the subject. We will start with the chemical, CVD, methods and recall some properties.

In India they synthesized graphene by using thermal decomposition of ethyl alcohol C_2H_5OH as a source of carbon with $NaBH_4$ and $NaOH$ as factor assistants for the interaction. The synthesis took place at a medium pressure in a stainless

steel chamber and the reaction was carried out for 20h at 230C°. After getting the product it is dried for 10h in a hot oven at 60C° (Singh, 2011).

This method has many advantages and disadvantages such as:

Disadvantages: - It takes a long time. It requires 20 hours in the initial step and another 10 hours for drying. Moreover, the products contained impurities like amorphous carbon, graphite, fullerene and some iron particles.

Advantages: - This method is a good way for bulk production. It is also a safer and commercially available process. Ethyl alcohol, as a source for carbon, is a cheap substance so we will use it as a source in our experiments, too (Singh, 2011).

The Japanese in the same year used another CVD method (Ayhan M. E., 2013). They synthesized graphene on Ag foil by using atmospheric pressure (AP). Chemical vapor deposition is done by using the solid camphor (which is derivative of $C_{10}H_{16}O$ as carbon source, or precursor in a gas which is a mixture of H_2 and Ar gases. Ag foils are heated up to 940C° in a tubular furnace (oven). The pure solid camphor was slowly evaporated and added into the growth zone along with a gas mixture (Ar + H_2). After that they cooled the furnace (oven) slowly down to the room temperature after coated with graphene. The foil was studied by introducing sulfur vapor in atmospheric conditions. So the pure sulfur was vaporized and the samples were exposed to sulfur vaporization for 15 minute period. This method also has advantages and disadvantages: **The advantage** is that, in this method (CVD) processes are similar to the tarnish-resistant coating which is commercially available. **The disadvantage** is the price of Ag foil. I was inspired by this method. I will try to find and use a cheaper material like Copper as substrate instead of Ag foil.

After some years, Japanese and Chinese used another method (Chen X. Z., 2015). They produced a 5mm hexagonal single-crystal graphene from ethanol by using thermal CVD system. In this system they used a quartz tube as the reaction chamber and copper foil as the substrate. Copper foils were cleaned and loaded into the CVD chamber. Reaction was to be at the center of the furnace (oven). The system was vacuumed to 25pa by using a mechanical pump for 10 minutes.

After that they heated the chamber to the reaction temperature (1065 C°) with Ar flow (300 Scm). Under the pressure of 300 pa, they replaced Ar flow by (300 scm) flow of 3% diluted H₂. Then, they sent ethanol vapor into the system to start the growth. This method requires about 2 to -24 hours. Some points we can see in this method as **advantage** are:-

- 1- It was low defect.
- 2-The outcome was homogeneous.
- 3-The single crystals which they grew was pure graphene.
- 4- Single crystals were as large as (5 mm).

Yet, one of the **disadvantages** in this method was the required time which was very long (2 to 24 h). Planned some aspects of my research based on this method such as using ethanol as the source of carbon, the quartz tube as the reaction chamber and copper foils for substrate.

So far we focused on CVD methods. From this point on, I will talk about physical methods which are actually a mixture of both chemical and physical methods.

I want to start with the method from United States for graphene growth (Xu, 2007). They used chemical vapor deposition enhanced plasma (**PECVD**) process which was carried out at 650C° using CH₄ as the source of carbon. They used a sample of Cu as substrate and the sample was put inside a 6-inch diameter quartz tub. The quartz tub was placed inside the furnace. By using RF capacitive plasma (13.56 MHz), after loading the sample, the chamber was pumped /completely evacuated and then heated to 650C° that took 20 minutes with flow of Ar gas. To start the growth, CH₄ was injected into the system. When the temperature reached the set point after the growth period, CH₄ flow was turned off and the system was cooled down. There are some **advantages** in this method. One is the short time required for production. Moreover, the procedure of one-step and after the rapid plasma enhanced CVD process, it was homogeneous. I like this method so I will take or use some parts of it such as using furnace (oven) and there hollowed in the center of oven the quartz tube placed in, inside the quartz tube they put or used Cu as substrate. However, I

will change the source of carbon with another chemical compound. Therefore I will use plasma but not capacitive.

Lastly, I want to provide an overview of the methods or growth or synthesis of graphene developed in Singapore (Lim S. C., 2004). Ethanol vapor is used as carbon feedstock with inductively coupled plasma (ICP) RF coil which was connected to (13.56 MHz) and was cooled with water. The plasma power was (250 W). The substrate was Cu foil that was put on a boat. The boat was placed in the center of the quartz tube. Furnace and the last (tube) was (21 cm) away from the ICP coil. They used a vacuum pump (0.1 mbar) to remove gases from the chamber. After that, they sent Ar gas to flow inside the tube. When the temperature reached the point (875 °C) they inserted ethanol vapor for the growth. This was carried out for 30 min and then they cooled the reactor. I think this method was ideal. Therefore, I will try to use the same method with some modifications. I will use inductive plasma but I will change (13.56 MHz) with another power supply and another matching device at (40.68 MHz). I will use another parameter for the inductive coils which surround the (2-inch) quartz tube and I will change the number of laps profile or coil number from 5 to 3. Then I will compare the results from this method with my modified to the method.

3. EVAPORATION AND CHEMICAL VAPOR DEPOSITION (CVD)

ENHANCED PLASMA

3.1. Introduction

Before delving into this subject, we must explain some Concepts that will be useful for understanding it such as concept and principle of evaporation and chemical vapor deposition (CVD).

3. 2. Defining Evaporation

Evaporation can be defined as the process where liquid is transformed into gaseous state (Institute for Atmospheric and Climate Science, 2016)

So, it is a kind of vaporization of a liquid that happens from the surface of liquid to a vaporous stage or gas phase. It is not saturation phase with evaporating essence or content. There is another kind of vaporization which occurs through boiling. It is characterized by formation bubbles of saturated vapor that occurs in the liquid phase. The steam produced; in boiler is another example for evaporation. It happens in a saturated vapor stage. Another kind of evaporation occurs straight out (directly) of the solid phase which is below the melting point. We can observe the ice below freezing temperature can turn into vapor speedily with enough heat. In fact, it depends on boiling point. When particles collide, the energy transfer to each other in different or varying degrees depending on the way they collide. Sometimes after the collide the energy causes molecules (particles) to move in one direction near the surface which enables molecules with enough energy to escape from liquid to the gaseous state (Silberberg, 2006). Evaporation may occur in low pressure as in our experiment, when the pressure is reduced by rotary, the liquid that we use (ethanol) as a source of carbon for the synthesis of graphene is evaporated. Ethanol /'εθənl/, or simply alcohol C_2H_5OH , C_2H_6O or $EtOH$, is widely used in alcoholic beverages. Produced by the fermentation of sugars by yeasts, Ethanol is a volatile, flammable, colorless liquid with a slight chemical odor. It is used as

an antiseptic, a solvent, a fuel, and, the active fluid in post-mercury thermometers (due to its low freezing point) (Shakhashiri, 2009)

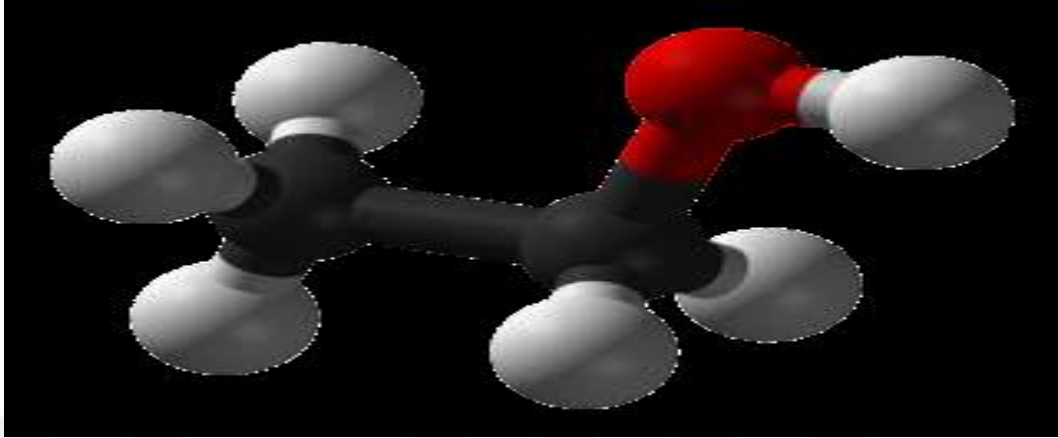


Figure 3.1. Chemical structure of Ethanol. (Ivan, 2010)

3. 3. Principe of Evaporation

The principle of evaporation is simple for coating material which is known as source material heated at low pressure ($\approx 10^{-3}$ pa) to above its boiling point. After that atoms and particles are introduced to distribute it by trajectories (tracks) in straight line to the substrate. Then these condense to form a thin film (Pierson, Hugh O., 1999).

There is a note it must be taken into account that most of the process known as reactive evaporation uses the nonmetallic element of the coating like carbon it is introduced into the gas phase and a pure metal source is used so the evaporation occurs in hydrocarbon atmosphere (Pierson, Hugh O., 1999)

3. 4. CVD

Chemical vapor deposition (CVD) can be defined as the process of deposition from vaporization or the deposition of a solid material on a heated surface and that are resulted from a chemical reaction for material in the vapor phase (Pierson, 1992)

So, it was received on solids as materials after vaporization material and converted it into a form as a coating , powder or as single crystals, by changing the characteristics of the material conditions or experimental of the substrate and the substrate temperature formation by the reaction gas mixture ,the total gas pressure flows , etc. with various materials it can be grown properties , there is featured characteristic of an advantage of the CVD technology is its excellent throw power or energy , allowing the production coatings of uniform thickness and properties a low of porosity on substrates of complex form , anther properties it is the possibility of localized or selectivity deposition on patterned substrates ,so CVD is woks in many application of thin film (Peter_M._Martin, 2010) .

(CVD) technology is a widely used its used materials-processing technics most of its applications includes applying solid thin film coatings for surfaces (Pierson, 1992).

The CVD technology is also used to produce a high-purity bulk materials and powders. Moreover.

The CVD are used in many application in industry and it has a larger – scale studies as a parts of CVD- larger treatments of thin film deposition (Ho, 2001)

In many aspects, the CVD competes directly with the PVD processes. Meanwhile, it is also used in conjunction with them and many of the newer processes are actually combinations of the two systems such as plasma enhanced CVD or activated sputtering processes. Public knowledge of the PVD processes is very important for the full understanding and appreciation of CVD (Pierson, 1992).

3. 5. PECVD

Nowadays, the most widely used method in IC fabrication is the PECVD, which means plasma enhanced chemical vapor deposition. This is due to fact that the dimensions have decreased and imposed the condition for more stringent or compact tolerances on the dopant location in depositing films. The last point we illustrated, was the chemical aspects of plasma which are usually of utmost

importance. But the physical effects of plasma can be important for determining the properties of films fabricated by PECVD methods.

This method is still used in its simplest form from the earlier days to present. PECVD requires increasing the temperature of the substrate to a required value in order to achieve the requirements of the chemical reaction to take place in a controlled manner (Rossnagel, Cuomo, & Westwood, 1990).

In a PECVD reactor a number of intertwined processes occur, which can be simplified by the following scheme:

- 1- A glow discharge is initiated by the ionization and dissociation processes by electron impact.
2. The second stage plasma is driven by diffusion to the wafer surface.

The gas phase reaction and transport of the reactive species.

3. The third stage is the adsorption of reactive species on the wafer surface sites and the reaction of these species with surface atoms or with other adsorbed species to form a reaction product.
4. The fourth stage is surface diffusion of the adsorbed species and reaction products. Nuclei grow into island-like piles and these merge into a continuous thin film.
5. The fifth stage is desorption of the volatile reaction products followed by transportation of products away from the growth region.

It must be noted that ion bombardment aids processes 3, 4 and 5. The quality of the growing films depends on the percentage of different kinds contributing to the growth that is very important. Also the deposition prices or costs are important (Boogaard, 1971).

While these reactions are well understood, they require exact high temperatures in PECVD methods so the chemical effects of the plasmas allow the reactions to proceed at lower temperatures that is needed to prevent spreading or the diffusion of the dopant materials during the next or subsequent processes (Rossnagel, Cuomo, & Westwood, 1990).

The depositions of films at different temperatures are determined by plasma environment and the heat is generated by the reaction. While no high temperatures are needed for the chemical reaction when we use plasma, the properties of films may be improved by heating (Rossnagel, cuomo, & westwood, 1990).

In our experiment, by a typical reactor for PECVD is low-pressure plasma chambers of a few liters in Volume, ethanol vapor diluted was passed with other precursors and argon, which it pumped at a certain flow rate. A gas mixture enters the reactor at room temperature. For film deposition a substrate is located in the chamber so the substrate can be part of the electrical circuit that drives the discharge (direct PECVD) or is electrically separated from the electronic circuit that generates the plasma (remote PECVD). An RF oscillating voltage is applied to an electrode inside or outside the chamber. In our experiment we used the latter configuration (Rossnagel, cuomo, & westwood, 1990).

4. GRAPHENE

4.1. Introduction

Graphene can be defined by its properties. It is a two-dimensional (2D) sheet (Ayhan, Kalita, Sharma, & Tanemura, 2013), with a single atomic layer thickness (Singh, Dilip Kumar, IYERY, & GIRI, 2011). It is a zero band gap semiconductor which is characterized by massless charge carriers that have sp^2 – plane bonded to carbon atom structure (Viet Phuong Pham, 2015). The bonds of carbon atoms are densely packed in a honeycomb crystal lattice (Singh, Dilip Kumar, IYERY, & GIRI, 2011). Graphene has extraordinary properties such as very high carrier mobility, high electrical and thermal conductivity, high optical transparency, high chemical stability, and in addition it has a high mechanical strength, etc.

According to all properties, graphene is considered the next generation of semiconductor. So, it is the display devices to replace the current device technologies (Viet Phuong Pham, 2015). The monolayer of graphene or single-walled carbon nanotubes can be grown by mechanical exfoliation such as CVD method enhanced by plasma for large-area monolayer graphene on transition metals foil like cobalt, nickel and copper as a catalytic layer in order to obtain a high-quality graphene growth (Viet Phuong Pham, 2015). The best one of these transition metals is copper (Cu) (Ayhan, Kalita, Sharma, & Tanemura, 2013). Copper is used on SiO_2/Si substrate, PECVD for graphene processing.

So PECVD single – walled carbon processing involves thermal CVD. This method generally contains graphene growing on a catalyst surface like Cu, by using a mixture of a hydro-carbon gas precursor or any carbon feedstock's, such as hydrocarbon, volatile organic solvents or carbon monoxide, which adsorb on very small catalytic particles. So at high temperatures (~650-1150 °C) chemical compounds of carbon feedstock are decomposed to generate atomic carbon species. So these atomic carbon species are organized themselves into nanotube forms. All this happens in the presence of small catalytic particles. But, the T-CVD hasn't achieved optimized single – walled carbon chirality control yet. This

is because it is reason is difficult to precisely control the decomposition of carbon feed stocks (Li Wei, 2013).

The plasma process plays a very important role in the manufacture and development of microelectronic devices. Plasma based on the single – walled carbon (graphene) synthesis method is attractive because of the possibility to achieve better control and to integrate single – walled carbon synthesis with electronic device manufacturing (Li Wei, 2013).

Actually there is a few studies that seek to explain the potentials and underlying physical processes of plasma which depends on the methods for chirality-controlled single –walled carbon synthesis such as selective nucleation etching, preparation of well-defined catalysts and reacting tubes after growth (RM, 2011) (K. Ostrikova *, 2013).

The process based on micro-plasma has been used to structurally controll metal nanoparticle and to prepare size- for SWCNT growth (Chiang WH, 2009). So, the Plasma enhanced CVD (PE-CVD) can enhance the decomposition of carbon feedstock at a temperature much less than (350–800 C) compared with T-CVD (Kato, Jeong, Hirata, Hatakeyama, Tohji, & Motomiya, 2003). In early times the PE-CVD studies generally achieved multi-walled carbon nanotubes or carbon fibers (Meyyappan M. , A review of plasma enhanced chemical vapour deposition of carbon nanotubes, 2009) (Meyyappan, Delzeit, Cassell, & Davi, 2003), so the direct plasma etching have small damages, which is characterized by small diameter single – walled carbon.

With the optimization of PE-CVD growth parameters, it may lead to the formation of SWCNTs (Bae , Min , Kang , & Ko, 2005) (Min , Bae , Oh , Kang, & Park, 2005), and even single – walled carbon with narrow chiral distribution (Zohreh Ghorannevis, 2010) . Remote PE-CVD (RPE-CVD) is a method which puts the plasma source far away from the site of SWCNT growth. Because of this interferences or damages by plasma toward single – walled carbon (graphene) growth can be reduced to a minimum (Guangyu Zhang, 2006).

There are many studies for PE-CVD for grapheme synthesis. But the best one is single – walled carbon chirality as single – walled carbon chirality can be also

changed during the process of restructuring for carbon caps that include the asymmetric defects (Neyts, van Duin, & Bogaerts, Changing Chirality during Single-Walled Carbon Nanotube Growth: A Reactive Molecular Dynamics/Monte Carlo Study, 2011) (Neyts, Ostrikov, Han, Kumar, van Duin, & Bogaerts, Defect Healing and Enhanced Nucleation of Carbon Nanotubes by Low-Energy Ion Bombardment, 2013). This process for defect healing is affected by the continuous addition of new carbon atoms, which can be controlled by PE-CVD (Neyts, van Duin, & Bogaerts, Changing Chirality during Single-Walled Carbon Nanotube Growth: A Reactive Molecular Dynamics/Monte Carlo Study, 2011) (Neyts, Ostrikov, Han, Kumar, van Duin, & Bogaerts, Defect Healing and Enhanced Nucleation of Carbon Nanotubes by Low-Energy Ion Bombardment, 2013). This way RPE-CVD work as a potential tool in the generation of the best suitable carbon radicals from carbon feedstocks. For this reason, it is very useful to explore the prospective of RPE-CVD in chiral selectivity for growth of SWCNTs.

Plasma Enhanced Chemical Vapor Deposition Symbolized by the (PECVD) enables lowering the temperature as the necessary energy for breaking the precursor molecules is provided through the plasma production, there are many reports about PECVD growth of graphene at low temperatures (Kim,J., Ishihara,M., Koga,Y., Tsugawa,K., & Hasegawa,M., 2011) (Muñoz, R. & Gómez-Aleixandre, C., 2013).But, most of these work so far have focused on catalytic approaches and little interest has been given to arbitrary non-catalytic substrates. The previous few attempts of growing graphene on noncatalytic materials either needed a long time up to several hours to obtain full coverage of the growth (Zhang, L., Shi, Z., Wang, Y., Yang, R., Shi, D., & Zhang, G., 2011) (Kim, Y. S., Joo, K., Jerng, S. K., Lee, J. H., Yoon, E., & Chun, S. H., 2014),This makes them commercially unviable. They also lack complete analysis for the expansion of graphene growth during PECVD (Medina, H., et al., 2012) (Muñoz, R. & Gómez-Aleixandre, C., 2013) .

Co incorporated MCM-41 catalyst (Co-MCM-41)² was used widely in many new method for graphene synthesis. The reason for this is its excellent performance in producing single – walled carbon with narrow (n,m) distribution (Lim S. C., 2003). It produces enriched single – walled carbon at temperatures (650–1150 C°) and carbon monoxide pressure at (6 bar) in T-CVD (Chen, Y., Ciuparu, D. , Lim, S., Yang, Y., Haller, G. L., & Pfefferle, L., 2004). For potential integration of single-walled carbon growth with electronic device manufacturing, it is very desirable to reduce the growth pressure and temperature, and to change the toxic carbon monoxide with a nontoxic carbon feedstock. Ethanol is widely used for growing high purity single – walled carbon (graphene) in T-CVD while hydroxyl is thermally decomposed ethanol is known to facilitate the removal of amorphous carbon (Maruyama, S., Kojima, R., Miyauchi, Y., Chiashi, S., & Kohno, M., 2002) . Graphene have also been successfully synthesized using ethanol PE-CVD (Suda, Y., et al., 2009) (Xu, B., Li, T., Liu, X. , Lin, X., & Li, J. , 2007) .

In our experiment, we have studied the synthesis of single – walled carbon on Co-MCM-41 catalyst by using ethanol in RPE-CVD. It combined the benefits from three axes or three sides

- (1) Ethanol is able to synthesize high purity single – walled carbon as a nontoxic carbon feedback.
- (2) Co-MCM-41 catalyst has good (n,m) selectivity control .
- (3) RPE-CVD ensures a lower graphene growth temperature by generating carbon radicals out of graphene growth locations.

There are two key growth parameters that influence the quality of graphene, the plasma power and growth temperature, which were also methodically studied

² MCM-41 (Mobil Composition of Matter No. 41) is a mesoporous material with a hierarchical structure from a family of silicate and alum silicate solids that were first developed by researchers at Mobil Oil Corporation and that can be used as catalysts or catMCM-41 consists of a regular arrangement of cylindrical mesopores that form a one-dimensional pore system. It is characterized by an independently adjustable pore diameter, a sharp pore distribution, a large surface and a large pore volume. The pores are larger than with zeolites and the pore distribution can easily be adjusted. The mesopores have a diameter of 2 nm to 6.5 nm and support catalysts.

In this experiment, we achieve growth of layer graphene at (650 C°) by using remote plasma-enhanced CVD on both non-catalytic and traditional catalytic substrates. Our experiment allows full coverage of graphene over large areas within only a few minutes. This work provides of grapheme growth rates on different substrates using PECVD. We use Raman spectroscopy, Scanning Electron Microscopy (SEM) and optical measurements to analyze the progression of graphene growth with time that is in sharp contrast with the scheme observed in thermal CVD. Our aim was also to provide an insight into the mechanism of low-temperature PECVD growth where there is no catalytic action is present. This approach provides a one-step method towards catalyst-free, and high speed growth of multilayer graphene at reduced temperatures (Chugh, S., Mehta, R., Lu, N., Dios, F. D., Kim, M. J., & Chen, Z., 2015).

4.2. Graphene (Single Wall Carbon)

A single layer of planar graphite sheet, which has six-membered rings of sp²-bonded carbon, atoms formed a two-dimensional network is called a graphene (Iijima, S., 1991).

So this rolled-up graphite was discovered by Iijima In 1991. The structure of graphene was called a carbon. When graphene was discovered, it was a multi-walled carbon (MWC), in which several graphite sheets were interlaced and it was formed in concentric cylinders. In 1993, a single-walled CNT, In 2004 Novoselov, Geim, and

Colleagues they try to peeled off the highly-oriented pyro-lytic graphite (HOPG) with adhesive tape and formed a few-layer graphene sheet on a substrate. This sheet exhibited quite high electron mobility, in the vicinity of 10,000 cm²/V·s (Chen, J. H., Jang, C., Xiao, S. , Ishigami, M., & Fuhrer, M. S, 2008). Because of good properties of graphene forthwith, it attracted wide attention. It was considered to have potential for application in electronic device fields where CNTs have been considered promising.

The first form of graphene transparent touch panel has already been created and the application development of graphene is being undertaken at high speed (Blake, P., et al., Making graphene visible, 2007). Hydrocarbon gas by plasmas attract attention because they can be used to synthesize large-area graphenes at low temperatures, which is called PECVD.

4.3. Properties of Graphene and Its Atomic Structure

In this section, we would like to provide a simple explanation. So, we describe below the properties of graphene. The atomic structure, thermal properties, optical properties, electronic properties and mechanical properties are included. We can see the shape of graphene structure in 2D hexagonal in Figure 3.2 and in 3.3



Figure 4.1. The shape of graphene structure in 2D hexagonal sheet of carbon atoms (Li Wei, 2013).

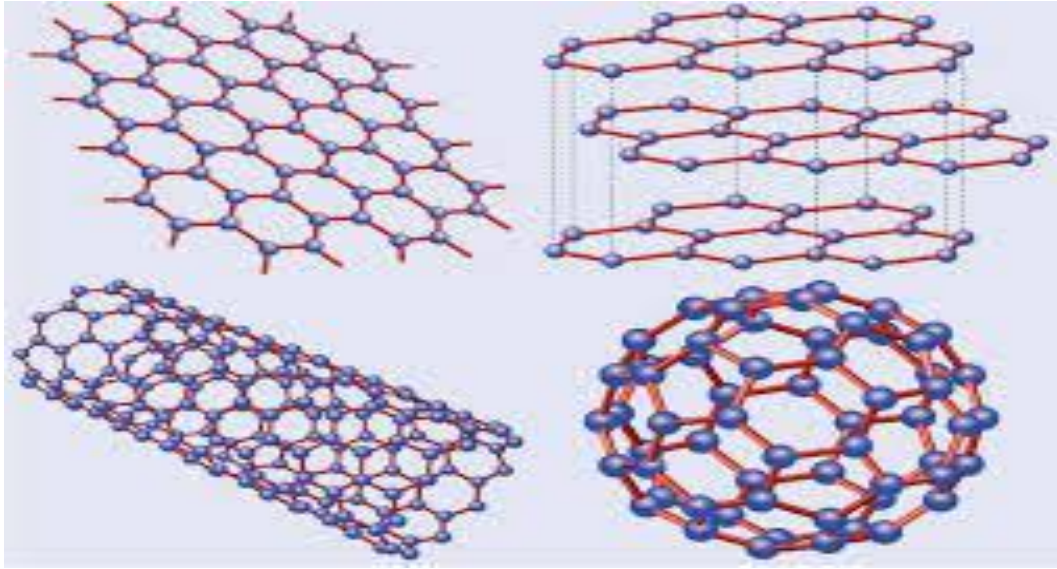


Figure 4. 2. We can see the shapes of graphene structure in hexagonal sheet of carbon atoms. (Li Wei, 2013)

As we noted above, the two dimensional structure of graphene is like a honey comb. The best way to study the structure of graphene is through a Transmission Electron Microscopy (TEM). It can also check the layers of graphene. Figure 4.3(a, b) shows how the graphene looks like under TEM. One is monolayer graphene and the other is bi-layer graphene. The difference between these two is their flowing edges, for a graphene in solid form.

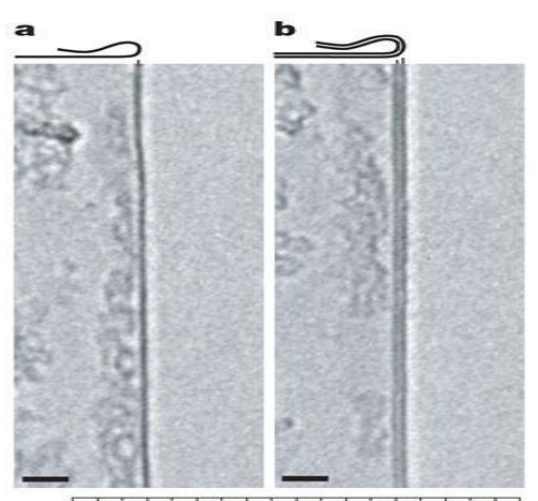


Figure 4. 3. (a) Monolayer graphene from TEM image5 and (b) Bilayer graphene from TEM image . Two images show different flow edges. (RM, 2011).

4. 3. 1. Graphene Electronic Properties

Graphene presents or shows as a two-dimensional (2D). It is like semi-metal with a zero band gap semiconductor (Novoselov, K. S., et al., 2007). It is characterized with massless charge a carrier that has Sp^2 -plane bonded to carbon atom structure (Pham, V. P., Kim, K. H., Jeon, M. H., Lee, S.H., Kim, K. N., & Yeom, G. Y. , 2015). Graphene exhibits an am bipolar electronic field effect (Zhang, 2013). Depending on the nature of graphene semiconductors, it must contain carriers for charge and gaps (Pham, V. P., Kim, K. H., Jeon, M. H., Lee, S.H., Kim, K. N., & Yeom, G. Y. , 2015). So we can say that depending on the nature of gate voltage, the charge carriers of graphene can be holes or electrons. The mobility of graphene was found to be $15,000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ or even higher (Novoselov, 2005) . Since there is a presence of defects or the corrugating surface of graphene, the mobility at room temperature is limited (Novoselov, K. S., et al., 2007). It is also found that the resistivity of graphene is lower than that of silver which is a well-known material having the lowest resistivity at room temperature (Chen, J. H., Jang, C., Xiao, S. , Ishigami, M., & Fuhrer, M. S, 2008) . Dirac-like equation is used to describe the charge carrier in graphene (Novoselov, K. S., et al., 2007). Through studies, scientists have found that graphene's crystal structure consists of two equivalent carbon sub-lattices. Two energy bands exist as a result of quantum mechanical hopping between the sub-lattices. The intersection near the edges of the Brillouin zone yields the conical energy spectrum near the "Dirac" points K and K (Novoselov, K. S., et al., 2007)'. Dirac point or so called neutrality point is the location where charge carriers change from electrons to holes. Two new quantum Hall effects raise the electronic properties of graphene. The quantum Hall Effect explained in quantum mechanics. It is observed in two dimensional electron systems which are under low temperature as well as strong magnetic field. In light of explanations above and all properties for graphene, it is considered the next generation of semiconductors. So it is the display device to replace the current device technologies (Pham, V. P., Kim, K. H., Jeon, M. H., Lee, S.H., Kim, K. N., & Yeom, G. Y. , 2015).

4. 3. 2. Optical Properties

The light transmittance property of graphene makes it to be a suitable candidate in optoelectronic devices. Graphene only reflects a small portion of the incident light in the visible region. Therefore, it is best for Solar Cells Industry technologies. If the layer increases, more incident light can be reflected (Blake, P., et al., Making graphene visible, 2007). High-speed optical pulses make inter-band excitation and this produces carrier population which is non-equilibrium in the valence and conduction bands (Breusing, M., Ropers, C., & Elsaesser, T., 2009). When the electrons-holes pairs in graphene are excited, electron-electron inter-band scattering and phonon emission will occur and a new Fermi-Dirac Distribution will be reached. On a Pico-seconds time-scale, the inter-band carrier relaxation and cooling of hot phonons emerge (Lazzeri, M., Piscanec, S., Mauri, F., Ferrari, A. C., & Robertson, J., 2005). Fluorescence cannot be observed in graphene as the relaxation time is very short. On the other hand, graphene can have luminescence when the band gap is induced.

4. 3. 3. Thermal Properties

The thermal conductivity of graphene was found to be extremely high compared to other metals such as silicon and copper. At room temperature, the thermal conductivity of graphene is larger than that of bulk graphite (Cai, W., et al., 2010). This can be explained by the Wiedemann-Franz law. It indicates that in thermal conduction, phonon is the dominant factor (Balandin, A. A., et al., 2008). Using some measurements like the near-room temperature, thermal conductivity of graphene was recently measured to be between $(4.84 \pm 0.44) \times 10^3$ to $(5.30 \pm 0.48) \times 10^3 \text{ W m}^{-1} \text{ K}^{-1}$. These standards or measurements, was taken through a non-contact optical technique, which is higher than that of measured for carbon nanotubes or diamond. Using the Wiedemann-Franz law, it can be shown that the thermal conduction is phonon-dominated. For a gated graphene sector, an applied gate bias causing a Fermi energy shift is much larger than $k_B T$. So it can cause electronic contribution to increase and dominate over the phonon contribution at low temperatures (Balandin, A. A., et

al., 2008). So, the Thermo-electric effects such as seebaeck effect, turning the temperature difference into electricity, can be found in graphene.

4.3.4. Mechanical Properties

Graphene appears as one of the strongest materials in the world. Its mechanical strength was measured by two methods. The first one is atomistic simulation method (Neyts, Ostrikov, Han, Kumar, van Duin, & Bogaerts, Defect Healing and Enhanced Nucleation of Carbon Nanotubes by Low-Energy Ion Bombardment, 2013); another one is nano-indentation technique (Novoselov, K. S., et al., 2004). It is reported that the steel, famous building material, is not as strong as graphene. In general, must be taken into account that the tensile strength of graphene reported as 130Gpa is ten times above that of steel (Novoselov, K. S., et al., 2004). Moreover, the Measurements have shown that graphene has a breaking strength 200 times greater than steel. These measurements were taken using an atomic force microscope (AFM). So, Chen et. Al, tried to prepare graphene paper and study the effect of thermal annealing (Neyts, Ostrikov, Han, Kumar, van Duin, & Bogaerts, Defect Healing and Enhanced Nucleation of Carbon Nanotubes by Low-Energy Ion Bombardment, 2013). The graphene paper was manufactured by graphene dispersions. By filtrating of certain amount graphene dispersion through an Anodisc membrane filter, the graphene paper can be obtained after air dried and peeled filter (Neyts, Ostrikov, Han, Kumar, van Duin, & Bogaerts, Defect Healing and Enhanced Nucleation of Carbon Nanotubes by Low-Energy Ion Bombardment, 2013). It reveals that graphene paper performs better in mechanical properties after thermal annealing because the better ordering of graphene stacks. In other words, the annealing makes enhancement of interlayer contact and interaction of graphene sheets (Neyts, Ostrikov, Han, Kumar, van Duin, & Bogaerts, Defect Healing and Enhanced Nucleation of Carbon Nanotubes by Low-Energy Ion Bombardment, 2013). These high values make graphene rigid and very strong. So, these properties could lead to using graphene for Nano-electro-mechanical systems (NEMS) applications such as in resonators and pressure sensors.

5. EXPERIMENTAL

5.1. Synthesis System

Graphene sheets were synthesized by the condition of chemical vapor deposition enhanced remote plasma (RPE-CVD) at low –pressure, by using the system as shown in Fig. 1. In our experiment, we generate inductively coupled plasma (ICP) at the entrance of a tube furnace. We achieved ICP generation by using water cooled copper inductive coils which was surrounding a (5 cm) quartz tube. (PECVD) system was set up by using 40.68 MHz 250 W radio frequency Rf power source through matching network to get maximum plasma efficiency, a quartz tube (145 cm length and 2.5 cm radius) and the oven that partially covered the quartz tube.

Feedstock's of carbon and carrier gases were injected through an axis symmetrical diffuser in one end of the chamber entrance. A boat that used to host catalysts was placed in the quartz tube at the center of the tube furnace, which was placed 20 cm away from the ICP coil. A thermocouple was attached to the inner surface of the oven cavity which surrounds the quartz tube, to monitor the temperature of reactor which contains catalysts inside the quartz tube. A vacuum pump was placed at the other end of the quartz tube to remove gases from the chamber. A needle valve was put to control the reaction pressure, and another various operation parameters like gas flow rate, temperature, and pressure and plasma power. It can be tuned to facilitate the synthesis of Single-Wall Carbon. A copper sheet was placed inside the quartz tube for graphene deposition. Ethanol vapor was sent into the 1 mTorr argon environment and power was turned on for plasma. The oven temperatures varied between 500-900 C°. The copper sheet surfaces were analyzed by Raman, AFM and SEM.

5.2. Single – Walled Carbon Synthesis

The graphene (single – walled carbon) is typically synthesized by using 80 mg of catalysts loaded in the boat of quartz. At first we used 650 C°.

The catalyst temperature was raised to the specific single - walled carbon (graphene) synthesis temperature. At that time, the reactor was evacuated by a vacuum pump to 0.1 mbar which is equal to 10Pa (Pascal). Later, the plasma system was turned on. Through graphene synthesis process, ethanol evaporated from warehouse at 2 mbar went through the plasma zone inside the quartz tube. Graphene growth was done for 4-5-6-7-8 minutes. At the end of the process, the reactor was cooled at room temperature under Ar flow. The experiments were repeated many times for each set of synthesis parameters to minimize system errors.

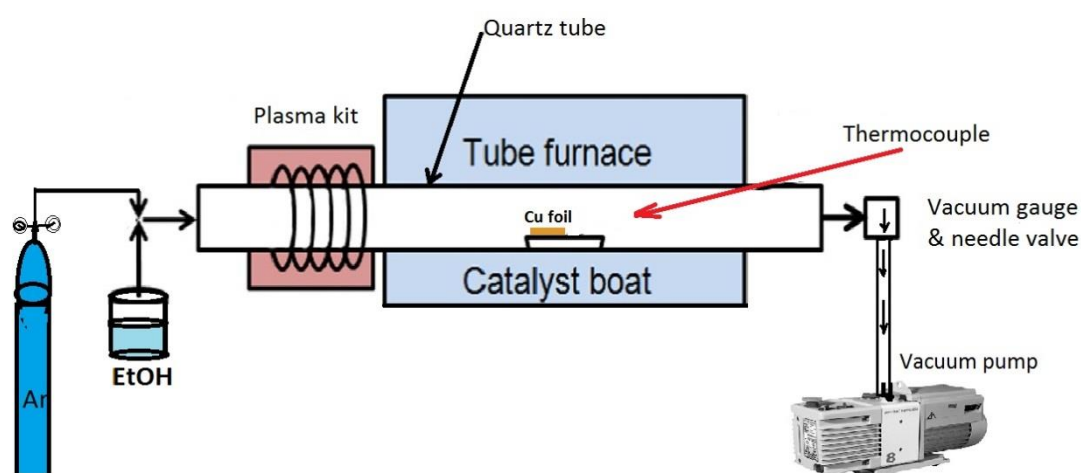


Fig.5. 1.A schematic drawing of the remote plasma enhanced CVD system.



Figure 5.2. By using 250W at the ICP view began to turn on the power before starting graphene synthesis work.

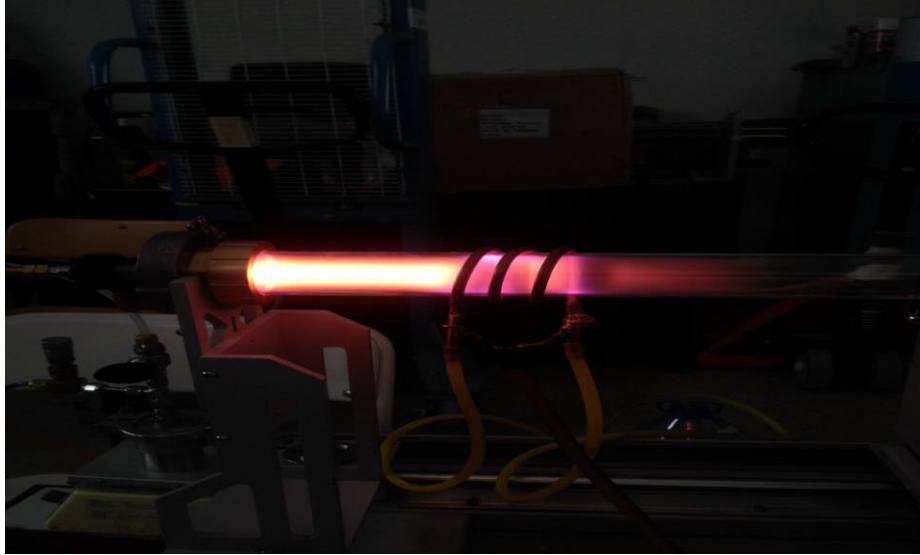


Figure 5.3. By using 250W at the ICP view began for graphene synthesis experiment enhanced plasma

5.3. Single - Walled Carbon Characterization for Graphene

In this study, the SWCNT characterization for graphene, focused on the purity and (n,m) distribution. Using atomic force microscopy (AFM), optic microscope (OM), Ocean Optics Spectroscopy (OES), Scanning Electron Microscope (SEM) and Raman spectroscopy, were used for the best evaluation for the purity of SWCNTs.

The intensity ratio between the SWCNT Raman disorder band (D-band) and the graphitic band (G-band) provide an indication of SWCNT samples purity. By using calculation the ratio of D/G (Itkis, M. E., Perea, D. E., Jung, R., Niyogi, S., & Haddon, R. C., 2005)

We calculated crystallite size using the Tuinstra and Koenig relation (J. Filik , 2005) (Wang, S. J., Geng, Y., Zheng, Q., & Kim, J. K. , 2010)

$$\frac{I_D}{I_G} = \frac{C\lambda}{L_a} \quad 5-1$$

Where

$$L_a = \frac{I_G}{I_D} C\lambda \quad 5-2$$

Where C stands for the pre-factor at 514.5 nm and equals to 4.4 nm and L_a is the crystallite size. The intensity of the D and G peaks were estimated by fitting the spectra. Values in above equation each represent amount where L_a is the crystallite size, $C(\lambda)$ is the laser wavelength used in the Raman system and C stands for the pre-factor at 514.5 nm and $C\lambda$ equals to 4.4 nm

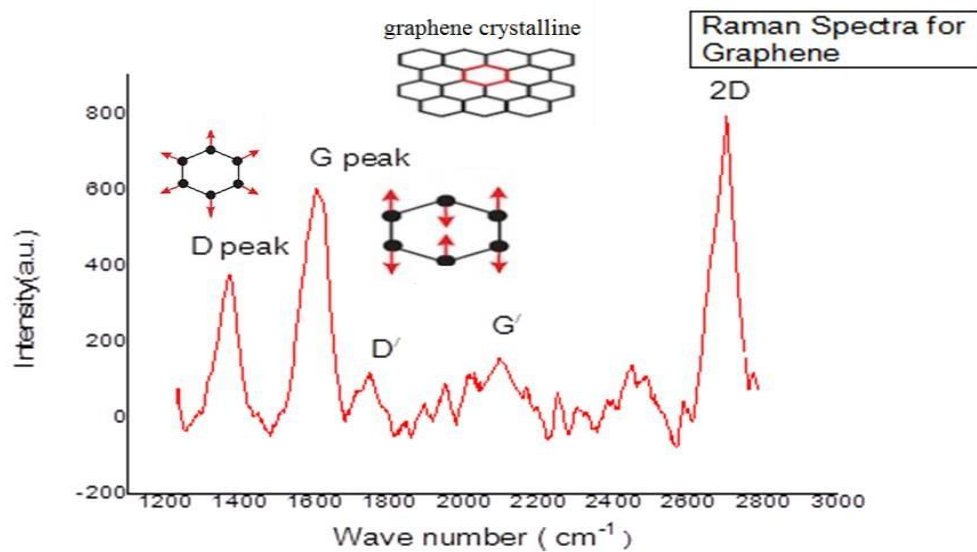


Figure 5.4. The 1598, 1 cm⁻¹ peak (called the G peak after synthesis crystalline graphene) the another peak (D peak), there is another peak like 2D, G/, D/ and we can see in over the Composition of a shape like bees crystal for graphene.

During Single-Wall Carbon (graphene) growth, we used (OES) to calculate and define the types of spectra emitted and the type and intensity of each element during growth. Then for the graphene growth, we directly use AFM to get the images which benefit us for getting a vision of growth. For determination, the synthesized carbon deposits were examined by a field emission SEM. After that, we used their Raman spectra collected by use of a Raman scope laser. The results were clear form which spectroscopic characterization was extracted.

We cleaned the copper (Cu) foils by some chemicals, such as ethyl alcohol and acetone distilled water. After that, the sample was first heated to 200 C°, and held for 10 min under air flow (200 sccm) to remove the humidity. And then, the temperature was increased from 200 C° to 650 C° at a 10 C/min rate. The weight loss of the sample was monitored and registered as a function of the temperature. The same procedure was repeated after the sample was cooled to room temperature for getting the second weight-temperature curve for baseline correction.

5.4. Synthesis of the Grapheme Layer

Graphene films monolayer was synthesized on Cu foil by PE- CVD method. Cu foil has an area of (3- 2.5 cm²) and the thickness of (14 mm) as seen in Figure2. Cu foil was rolled into a PE-CVD vacuum chamber made of quartz.

Before putting inside the furnace, the copper foil was cleaned by dipping in various solvents. The steps for these dips were

1. Acetone (10 seconds)
2. Water (< 10 seconds)
3. Acetic Acid (10 minutes)
4. Water (< 10 seconds)
5. Acetone (10 seconds)
6. Isopropyl alcohol (IPA) (10 seconds)

The remaining IPA was removed using compressed air. At First, the discharge chamber was filled with H₂ gas at a flow rate of 8 sccm , and the Cu foil was annealed for (~ 1 h) at a temperature of (625 C°) in an Ar environment. Next, graphene was synthesized at (650,725 , 850,950,1025 C°) in the environment of Ar/CH₃CH₂OH vapor (8/20 sccm) for 25 min, after which the chamber was cooled down to room temperature with Ar gas (10 sccm) for (~ 1 h). After the synthesis, the Cu foil was divided into small equally-sized pieces (~ 3 cm²). These small pieces of graphene on Cu foil were affixed on glass substrates using tape.



Figure 5.5. Photo for copper foil after graphene growth.

The glass substrates were used as holders or as a boat for the graphene – Cu foil assembly. In the case of pre-doped graphene, the plasma described below was doped directly on graphene Cu foil and then graphene for coating graphene which stood on Cu foil. For transferring graphene, there are many methods. We will explain our preferred method in the next section.

5.5. Transfer of Graphene

Transfer Process in our experience intended that the moment graphene was formed on a copper foil; it may be transferred on to any other substrates.

First, we used PMMA (4% in Anisole) which was drop-casted on the copper. This work took about 1.5 hours to evaporate. Following this, it was placed on hot plate at 145 °C for 3 minutes. A piece of scotch tape has been placed to the lower edge of the PMMA as a handle to be used later. After that, the sample was

cleaned by O_2 plasma for 2 minutes to remove the graphene from the opposite side of the copper. Then, it was put it in a ferric chloride ($FeCl_3$) bath overnight to remove the copper. Next the copper which was etching the sample was rinsed thoroughly in H_2O until ferric chloride Color could not be seen in the water. The graphene was stored in this way floating in a bowl of water. In order to transfer onto a substrate, the substrate was first cleaned. Then, the PMMA was placed on the substrate (graphene side down) directly after a bath of H_2O . Surface tension in the water held it on the surface carefully to avoid any wrinkles. The sample was placed in a covered acetone bath to remove the PMMA. The PMMA was then removed (with a bubble of acetone still covering the surface) and placed in an Isopropyl alcohol (IPA) bath for 1 hour. The sample then was put in a desiccator box in order to dry, and then it was ready for use.

We used spin-coated with PMMA for transfermaed graphene, while for the normally doped graphene, the grown graphene on Cu foil was spin-coated with PMMA without the chlorine doping.

For the pre-doped / un-doped graphene / Cu foil were dipped in a copper etchant solution (by using $FeCl_3$ solution) for 40 min on a hot plate ($40\text{ }^{\circ}\text{C}$) to completely remove the Cu foil. Then it was transferred onto PET or SiO_2 / Si substrates . SiO_2 /Si substrates were used for measurements for sheet resistance and optical transmittance.

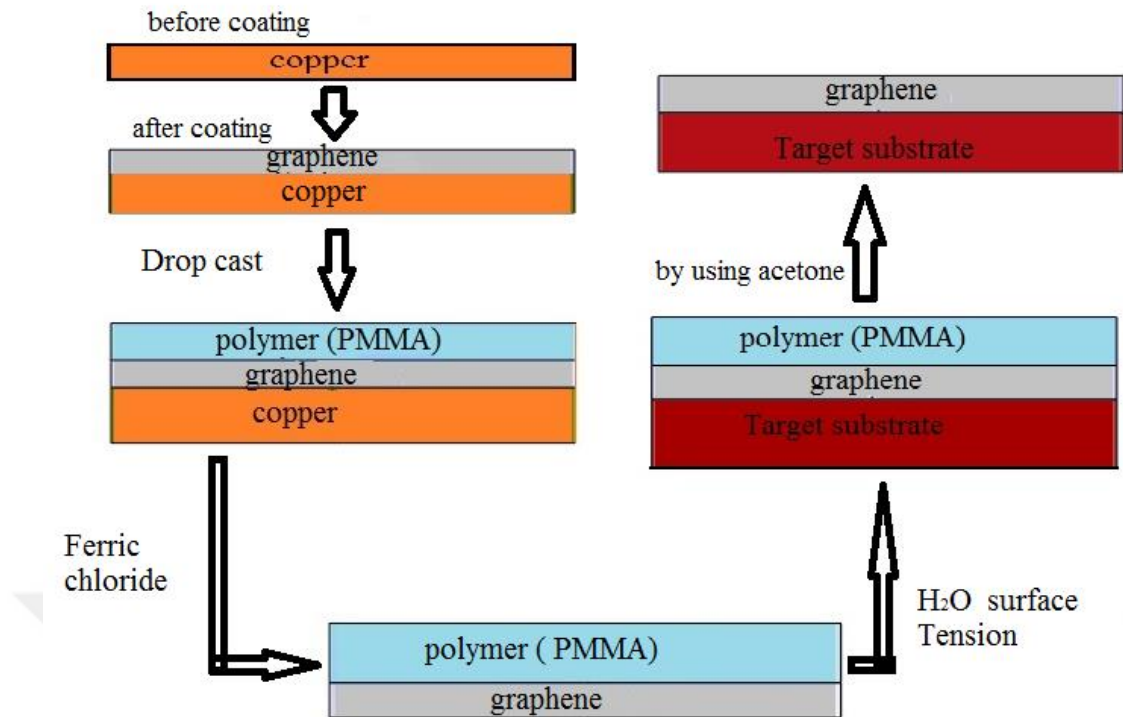


Figure 5. 6. Schematic diagram of graphene synthesis and the transfer process to an arbitrary substrate.

6. RESULT AND DISCUSSION

Table 6.1. The parameters and measurement for graphene Synthesis by PECVD method, first experiment.
Date: 26 – 02- 2015 at morning

Step	Parameters	measurement	
1-	Passage	2,9 *10 ⁻¹ Torr	-----
2-	Ethanol vapor	10 sccm	
3-	Argon	8 sccm	-----
4-	Matching	40,68 MHz	1000 watt
5-	RF	225 watt	-----
6-	Reflection	90	-----
7-	Time start the experiment by Ar	8.45----9.00	15 minute
8-	Time start oven to (T=650C°) +Ar	9.00----9.20	20 minute
9-	Oven in 650C° +Ar +plasma +Ethanol vapor	9.20-----9.23	3 minute
10-	Time stop and start cooling	9.23-----9.25	2 minute
11-	Weight of copper foil	Before experiment began 14 gr	After experiment began 16-17 gr
12-	The sample led us out and we started to stage a second then we analysis		

Note: The experiment failed and did not get a graphene nor on plasma. Maybe it was a failure because of the little time for graphene Synthesis and higher reflection for Plasma.

Table 6.2. The parameters and measurement for graphene Synthesis by PECVD method, second experiment.
Date:01 – 03- 2015 at morning

step	Parameters	measurement	
1-	Passage	1 *10 ⁻¹ Torr	-----
2-	Ethanol vapor	10 sccm	
3-	Argon	4 sccm	-----
4-	Matching	40,68 MHz	1000 watt
5-	RF	250 watt	-----
6-	Reflection	6	-----
7-	Time start the experiment by Ar	10.23----10.40	17 minute
8-	Time start oven to (T=650C°) +Ar	10.40----10.56	16 minute
9-	Oven in 650C° +Ar +plasma +Ethanol vapor	10.56-----stop	-----
10-	Time stop and start cooling	-----	-----
11-	The sample led us out and we started to stage a second then we analysis	-----	-----

Note: The experiment failed and did not get a graphene nor on plasma. Because of a disruption occurs in the cables of RF connected so we stopped the experiment.

Table 6. 3. Parameters and measurement for graphene Synthesis by PECVD method, thired experiment.
Date: 05 – 03- 2015 at morning

step	Parameters	measurement	
1-	Passage	1 *10 ⁻³ Torr	-----
2-	Ethanol vapor	10 sccm	
3-	Argon	4 sccm	-----
4-	Matching	40,68 MHz	1000 watt
5-	RF	250 watt	-----
6-	Reflection	4	-----
7-	Time start the experiment by Ar	8.50----9.05	15 minute
8-	Time start oven to (T=650C°) +Ar	9.05----9.25	20 minute
9-	Oven in 650C° +Ar +plasma +Ethanol vapor	9.25-----9.29	4 minute
10-	Time stop and start cooling	9.29-----	-----
11-	Weight of copper foil	Before experiment began 14 gr	After experiment began 13 gr
12-	The sample led us out and we started to stage a second then we analysis		

Note:- The experiment failed and did not get a graphene. There is a problem I am encountering it began or Starting from the third experiment, and for nearly seven experiments (3-----7). Due to burning of samples because a leak of oxygen gas accrues into the quartz tube.

Table 6.4. The parameters and measurement for graphene Synthesis by PECVD method, eighth experiment.
Date: 05 – 05- 2015 afternoon

step	Parameters	measurement	
1-	Passage	1 *10 ⁻³ Torr	-----
2-	Ethanol vapor	10 sccm	
3-	Argon	9 sccm	-----
4-	Matching	40,68 MHz	1000 watt
5-	RF	250 watt	-----
6-	Reflection	0	-----
7-	Time start the experiment by Ar	3.34----3.49	15 minute
8-	Time start oven to (T=750C°) +Ar	3.49----4.09	20 minute
9-	Oven in 750C° +Ar +plasma +Ethanol vapor	4.09-----4.13	4 minute
10-	Time stop and start cooling	4.13-----4.15	2 minute
11-	Weight of copper foil	Before experiment began 14 gr	After experiment began 16- 17 gr
12-	The sample led us out and we started to stage a second then we analysis		

Note: Experiment works or successful so we get a graphene enhanced plasma.

Table 6. 5. The parameters and measurement for graphene synthesis by PECVD method, ninth experiment.
Date: 24 – 04- 2015 at morning (We were delayed in the test and the reason because we were waiting for the results of Raman analysis of previous experience)

step	Parameters	measurement	
1-	Passage	$1 \cdot 10^{-3}$ Torr	-----
2-	Ethanol vapor	10 sccm	
3-	Argon	9 sccm	-----
4-	Matching	40,68 MHz	1000 watt
5-	RF	250 watt	-----
6-	Reflection	0	-----
7-	Time start the experiment by Ar	9.32---10.00	28 minute
8-	Time start oven to (T=850C°) +Ar	10.01---10.21	20 minute
9-	Oven in 850C° +Ar +plasma +Ethanol vapor	10.22-----10.28	6 minute
10-	Time stop and start cooling	10.29-----9.31	2 minute
11-	Weight of copper foil	Before experiment began 15 gr *4	After experiment began 16-17 gr
12-	The sample led us out and we started to stage a second then we analysis		

Note: The experiment was successful so we get graphene enhanced plasma. By conducting appropriate analyzes of samples. Like SEM, Raman, AFM and etc.

Table 6. 6. The Parameters and measurement for graphene Synthesis by PECVD method, ninth experiment.
Date: 05 – 05- 2015 afternoon

step	Parameters	measurement	
1-	Passage	1 *10 ⁻³ Torr	-----
2-	Ethanol vapor	10 sccm	
3-	Argon	9 sccm	-----
4-	Matching	40,68 MHz	1000 watt
5-	RF	250 watt	-----
6-	Reflection	0	-----
7-	Time start the experiment by Ar	3.47----4.17	30 minute
8-	Time start oven to (T=950C°) +Ar	4.18----4.38	20 minute
9-	Oven in 950C° +Ar +plasma +Ethanol vapor	4.39-----4.47	8 minute
10-	Time stop and start cooling	4.47-----4.49	2 minute
11-	Weight of copper foil	Before experiment began 15 gr *4	After experiment began 16-17 gr
12-	The sample led us out and we started to stage a second then we analysis		

Note:- The experiment was successful so we get a graphene enhanced plasma by PECVD method . So by conducting appropriate analyzes of samples. Like SEM, Raman, AFM and etc.

6.1. Effect of Plasma Power

After graphene was grown on copper foils by using the PECVD method according to the procedure we described above, we will discuss the characteristics of the produced material in this part. All the methods aimed primarily at verifying that graphene was indeed grown on the copper foil, and to get a sense of the uniformity of graphene coverage. The major purpose of introducing or using remote plasma in SWCNT.

Synthesis is to help the decomposition of carbon feedstock's, which depends on the plasma power (Meyyappan M. , 2009) . Before any action, we tried checking the first step. For this aim, the optical microscope images were taken for graphene that grown on copper foils at 300x magnification (see Figures 6.1 and 6.2). These images show three salient features.

First, the apparent regions as islands in figure 6.2. We can see two forms as islands, the small island on the left which was (0.543 mm^2) that is indicated by Green rectangular shape and on the other side of the same shape we can see a bigger island.

Secondly, the ridges in the copper substrate are clearly visible. They are a result of milling process of copper foils, as expected.

Thirdly, the weak or faint boundary lines can be seen separating regions of slightly different shade. Those are believed to be boundaries between domains of graphene growth Scanning Electron. Therefore firstly, we studied the effect of plasma power. The temperature inside the reactor was fixed at 650°C . Moreover the power of the remote plasma source was changed from 0 to 250W.

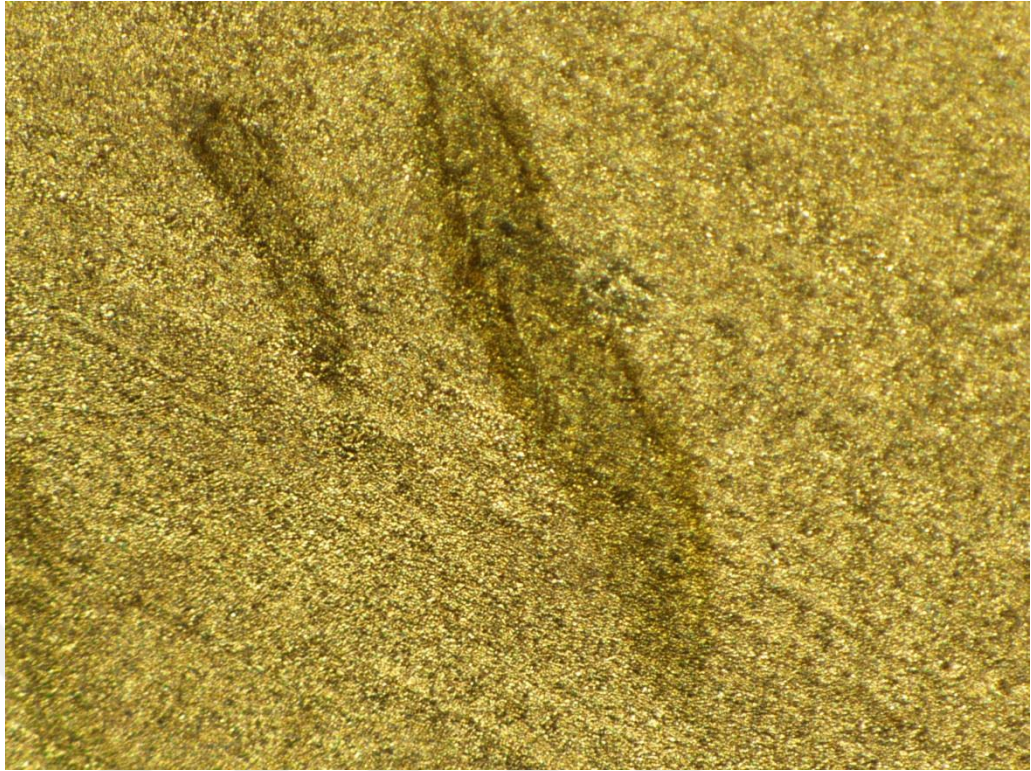


Figure 6.1: 300X Optic images. In the left Z: 0.0134 mm^Δ2 and right Z: 506 um

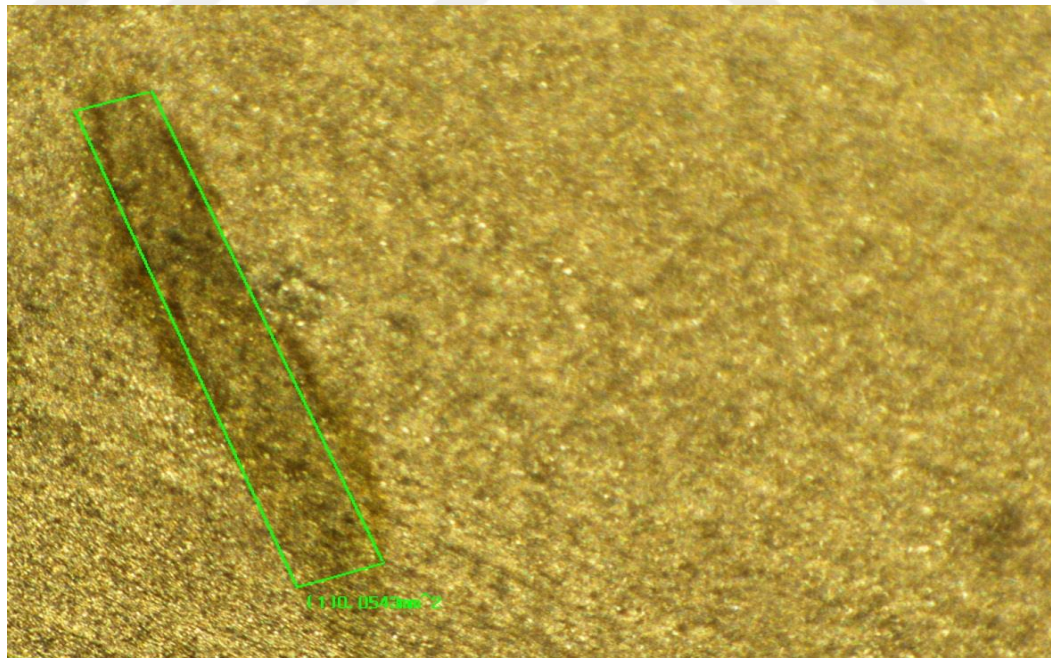


Figure 6.2. show 300X optic mag . Z: 543 um: Optical microscope image of graphene on copper foil.

Another method of analysis we use in our experiment as shown in Figure 6.3.



Figure 6.3. Ocean Optics HR4000 High-resolution model with 3 inputs (200-1100) nm spaced spectrometer

Ocean Optics HR4000 High-resolution is another method of analysis we tested, by using the optical emissions measuring that generated in the plasma we can distinguish Ocean Optics as shown in Figure 6.3, by using model HR4000 spectrometer's high resolution, optical fiber cables fixed to see the area where the plasma is formed.

The special constants were other data for atoms in the equation were in accordance with (NIST) which is established reference taken from the website.

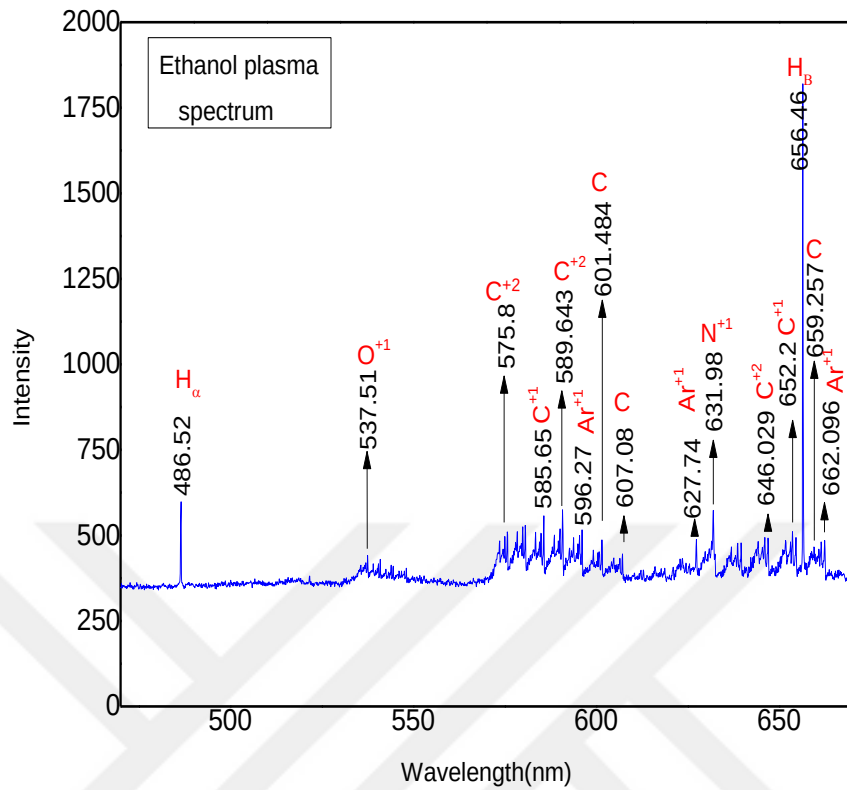


Figure 6.4. optical emission spectra obtained for argon plasma

By way of illustration for the symbols which measurements by Optical emission values and intensities obtained from argon plasma, so we obtain the data of atoms from (NIST) which is established reference taken from the website. So, we can recall them as electronic distribution of energy levels as seen in Figure 6.4. Optical emission spectrum has shown wavelength values transitions which are described below

Ion	Lower Level Conf., Term, J	Upper Level Conf., Term, J	Wavelength Air (nm)
C III	1s22p (2P°) 3d 1F° 3	1s22s6d 1D 2	575.811nm
C II	2s2p (3P°) 3p 4D 7/2	2s2p (3P°) 3d 4P° 5/2	585.606 nm
C III	1s22p (2P°) 3p 3S 1	1s22s5p 3P° 2	589.643nm
Cl	2s 22p 3p 3D 2	2s 22p 6s 3po 1	601.484nm
Cl	2s 22p 3p 3p 2	2s 22p6d 3po 2	607.839nm
C III	1s 22s 5p 1p° 1	1s 22s6d 1D 2	646.029nm
C II	2p3 2p° 3/2	1s26d 2D 5/2	652.264nm
C IV	1s26s 2s 1/2	1s27p 2po 1/2	659.257nm
Ar II	3s23 p4 (3P) 5p 2P° 3/2	3s 23p4 (3P) 7s 4P 3/2	596.137nm
Ar II	3s23 p4 (3P) 5p 4S° 3/2	3s 23p4 (3P) 7s 4P 5/2	627.74nm
Ar II	3s23p4 (1s) 3d 2D 3/2	3s 23p4 (3P2)4f 2[2] ° 5/2	662.0965 nm

Table 6. 7. Atomic Spectra Database Lines Data with wavelength for each contain from NIST

By using Scanning Electron Microscope (SEM) machine as in Fig 6.5

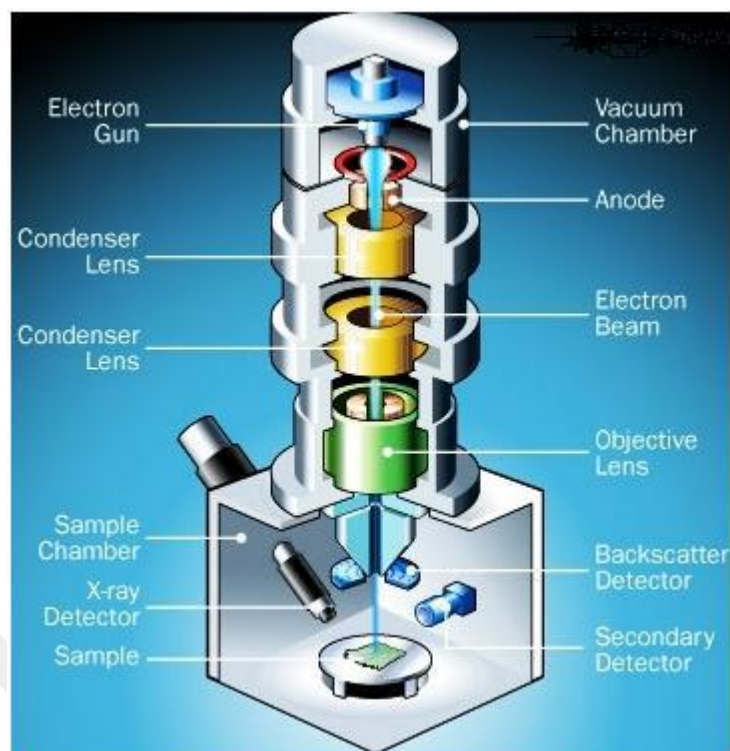


Figure 6.5: Schematic diagram of a scanning electron microscope (ATTEBERRY, 2009)

Initially we examined copper sample or foil before the experiment under Scanning Electron Microscope (SEM) as a reference's. As can be seen in figure 6.6.

The Scanning Electron Microscope (SEM) images taken for graphene grown on copper foils, illustrate many features of these samples. At low magnification (around 3.85kx, 4.09kx, 8.65kx, 19.62kx), copper domain boundaries are clearly visible as patches of varying shades (see Figures 6.7, 6.8 and in 6.9). These have been observed only on some samples. This may be due to a difference in copper annealing between the side facing up and the other side facing down in the furnace. At higher magnifications (8.65kx), we can note dark lines scattered across the surface of the sample (see Figure 6.8).

We believe that these are edges between the graphene areas; or maybe tears in the graphene resulting from a difference in thermal expansion between copper and graphene.

Also in Fig 6.7 at 3.85kx, the steps or ridges in the copper substrate can be seen. This is physical evidence that the graphene is covering the copper uniformly. In addition to that the copper would be oxidize in the furnace because it is cooled (and the steps would disappear) if the graphene was not there to protect it. Finally, black spots can be seen dotting the surface at 8.65kx (see Figure 6.8). It is unclear what these spots means. But, they seem to follow the graphene tears to a great degree. It is possible that these spots are partially oxidized copper that is exposed during breaks in the graphene. It is also possible, however, that these are blocks of graphite that are united or coalesced as the graphene formed. Finally, the gray spots that are shine on Figure 6.9 it is not clear what these spots it means are, but they seem to follow the graphene tears to certain degree .it is maybe these are blocks of graphite that united or coalesced as the graphene formed

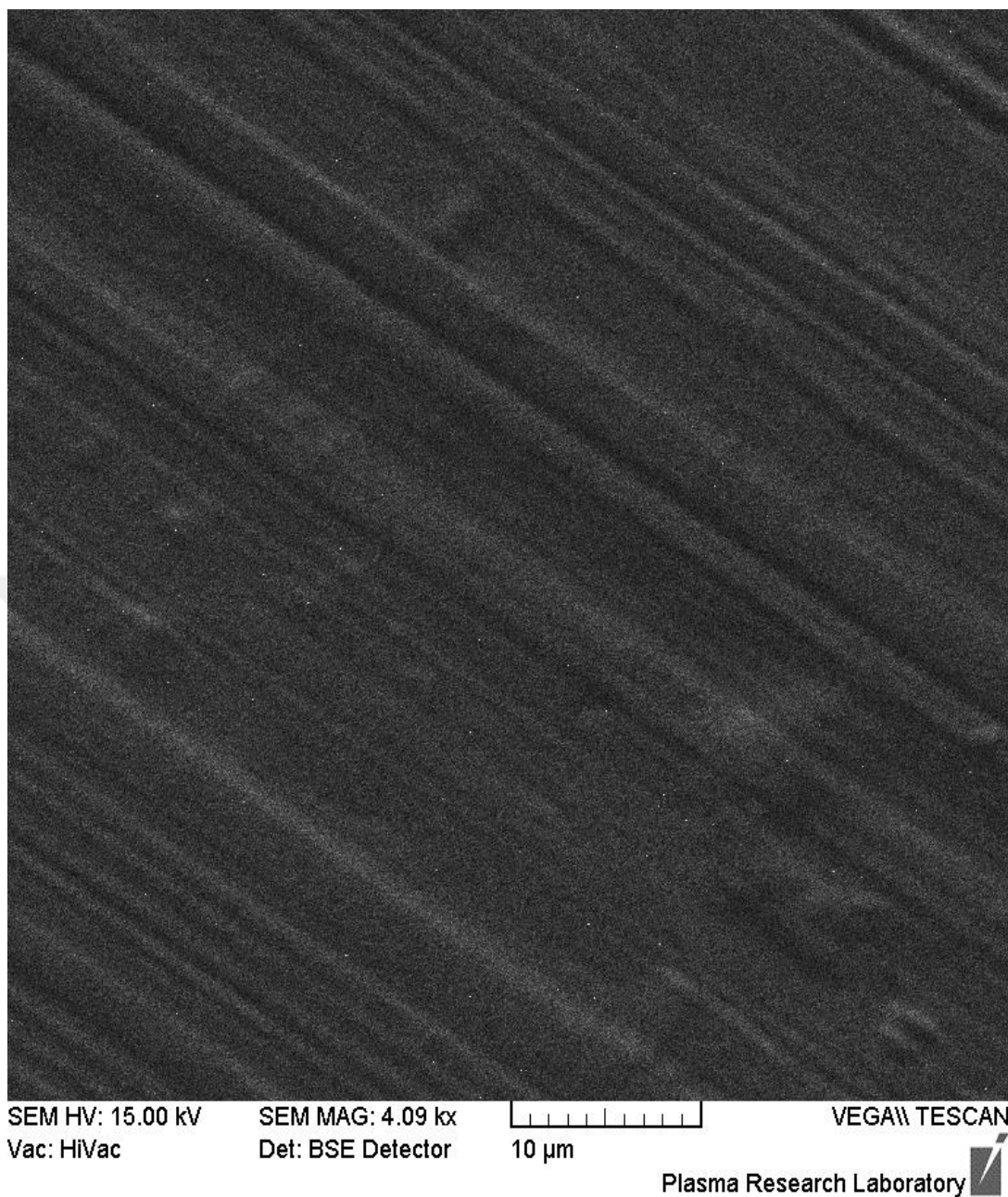


Figure 6.6. SEM image of copper domains, normal before syntheses graphene

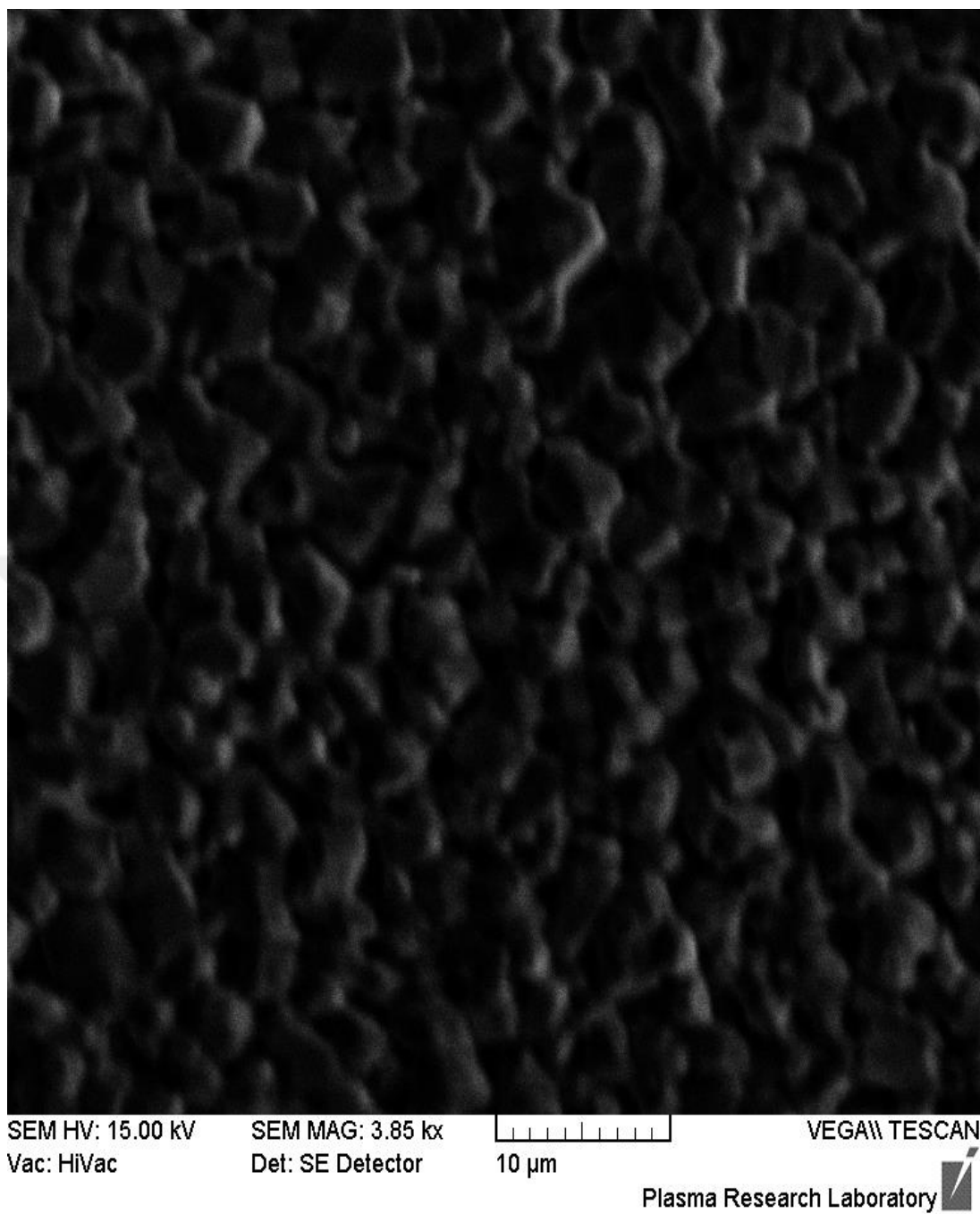


Figure 6.7. SEM image of synthesized graphene on Cu foil.in5 min

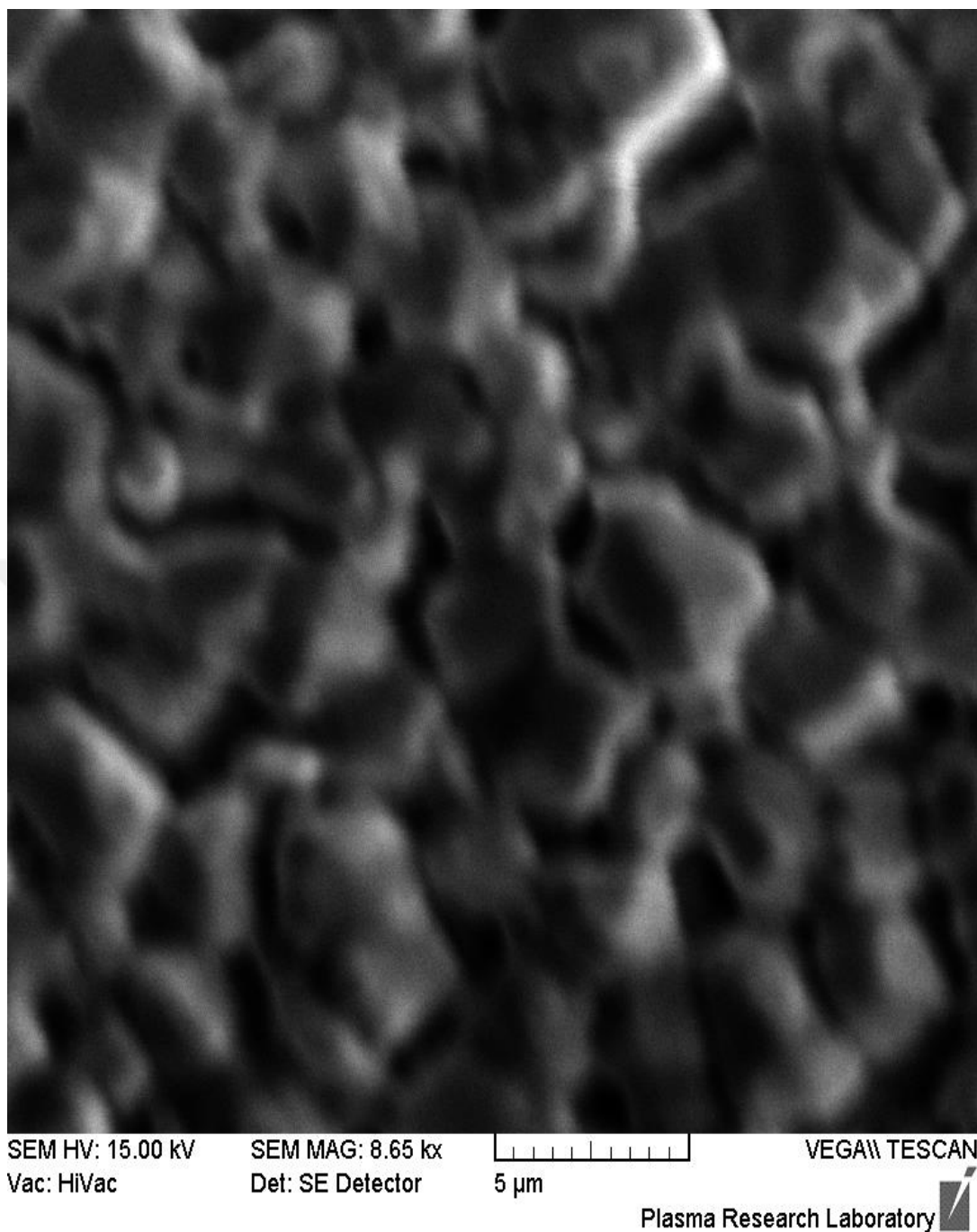


Figure 6.8. SEM image of synthesized graphene on Cu foil at 6 min

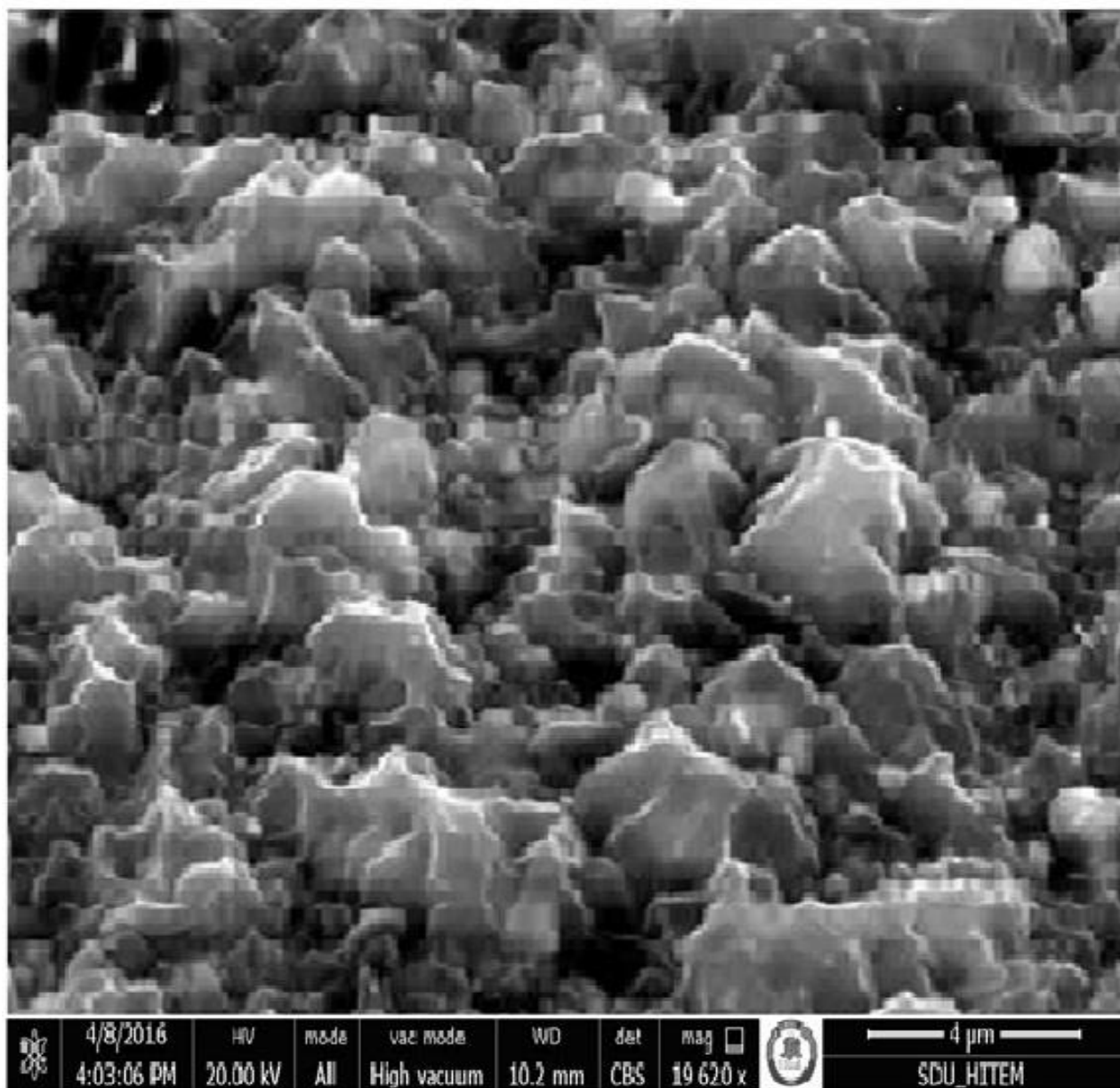


Fig 6.9 .SEM image of synthesized graphene on Cu foil. In 10 min

Atomic force microscopy (AFM) image were also taken, because it provides high-resolution images, as we know that the AFM is one of the most widely used tools for measuring, imaging and treating the samples on the nano scale. The AFM is used to scan the sample surfaces. In this technique, the measurements can be carried out at ambient environment without any special conditions.

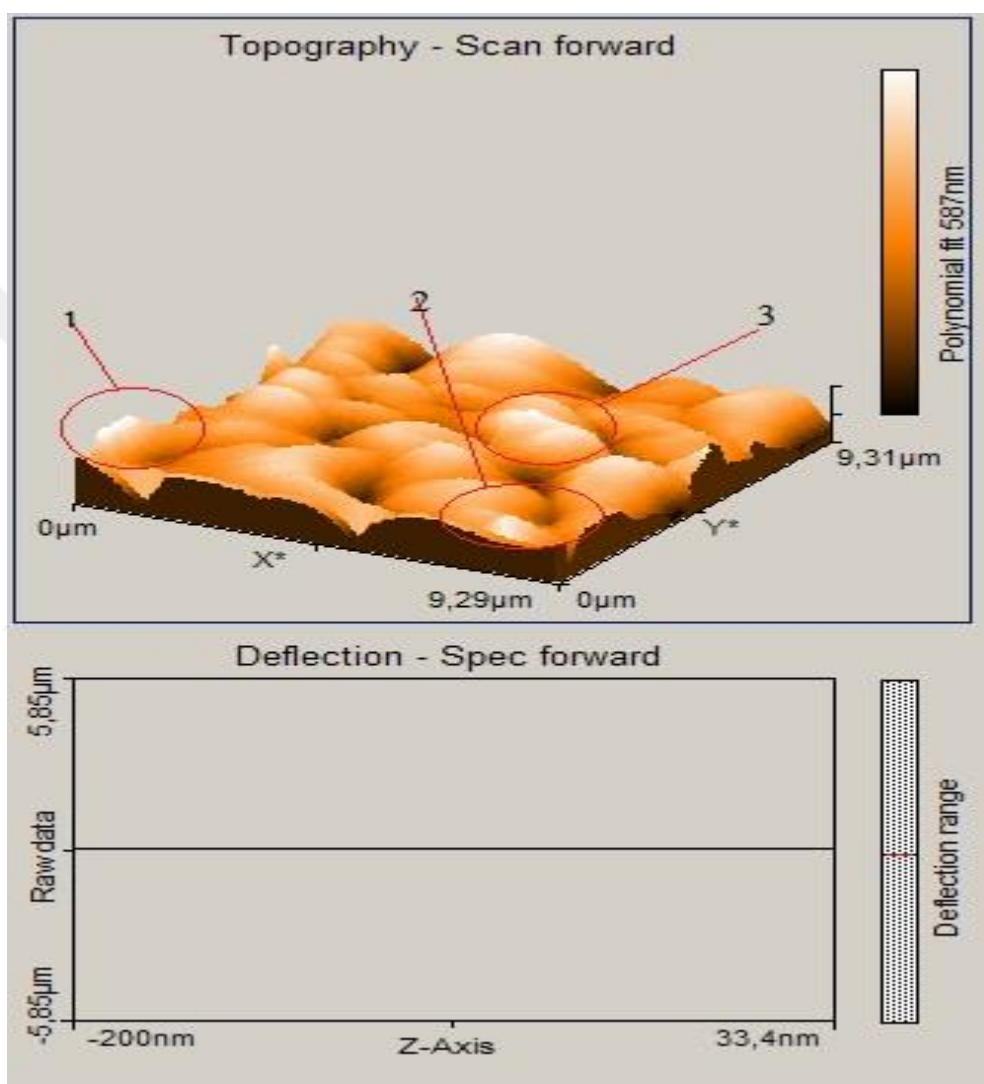


Figure 6.10. a. AFM image of sample Cu illustrates the position of graphene synthesis enhanced plasma.

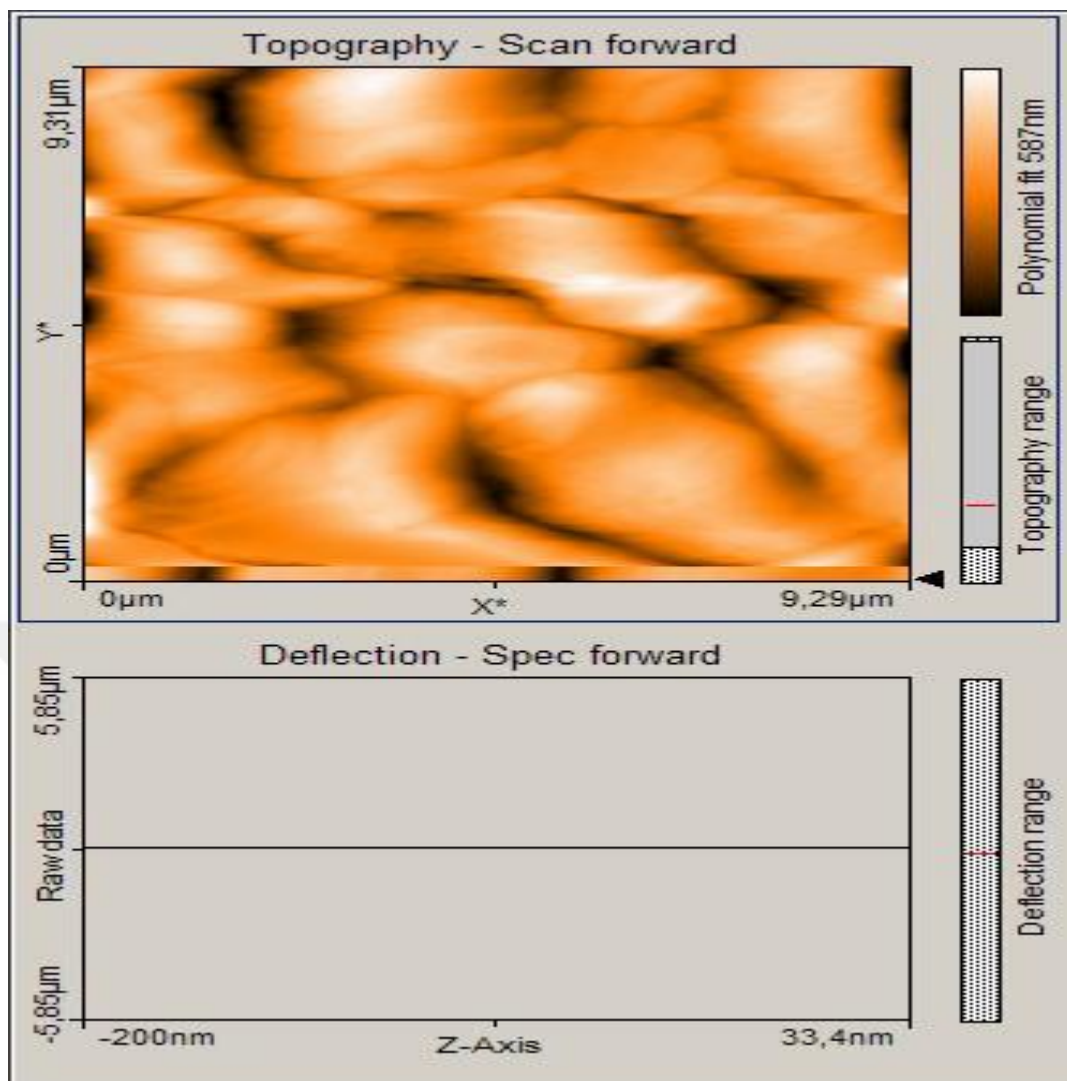


Figure 6.10. b. AFM image of sample Cu after graphene synthesis enhanced plasma from over.

The AFM studies were carried out in order to determine the thickness of graphene formation. The AFM images (Fig. 6.9 and in 6.10) show the type of graphene and that there are complex morphology that depends somewhat on the deposition conditions and regarding with thickness profile. The AFM images show a uniform thickness distribution with a height $\sim 9.29\mu\text{m}$ and $9.31\mu\text{m}$ with polynomial fit 33.4 nm .

But we cannot determine the number of the layers, especially in the case of graphenes synthesis from ethanol based solely on this data. Due to the fact that the wrinkled nature for graphene is like exfoliated, it cannot sit flatly on substrates. We believe that corresponding peaks which appear in Fig 6.11.a and

in 6.11.b are graphene layers. So, we used Raman studies after plot it as shown in the Figure 6.13 and in 6.14 for more certainty.

SEM images show that few single – walled carbon (graphene) exist on the catalyst at growth temperature of different carbon species.

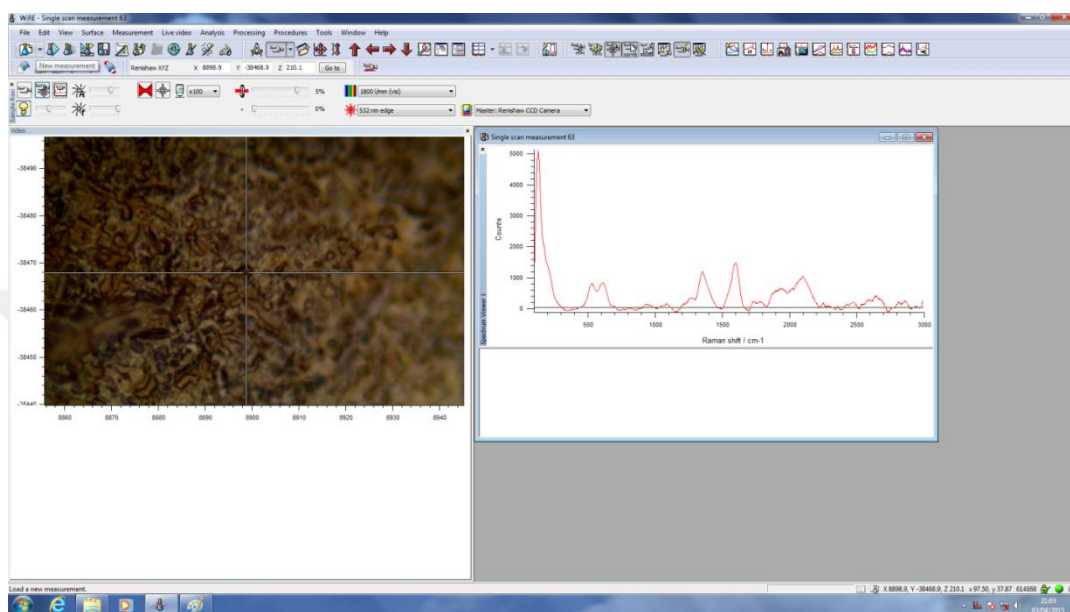


Figure 6.11.a. Raman spectra of graphene on SiO_2 , samples vacuum 10^{-3} Torr at 650 C° 4min.

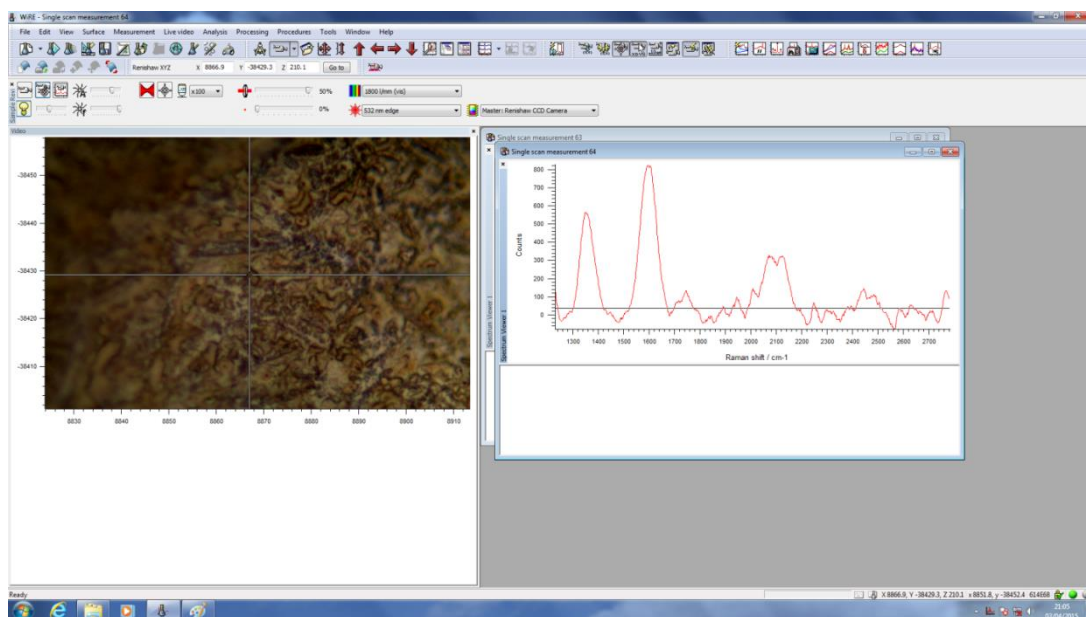


Figure 6.11.b. Raman spectra of graphene on SiO_2 , samples vacuum 10^{-3} Torr at 850 C° at 5min.

In order to ensure that graphene is correctly located on the copper foil, we plot the Raman spectra for grown graphene on copper as we can see in Figure 6.12 and after transferring graphene from copper we plot it again as seen in Figure (6.13 and in 6.14). Regarding the peaks in the graphene spectrum, we can see that they are different from the spectrum for the foil without graphene. So by using different conditions, all the experiments carried out show that plasma using ethanol as carbon feedstock is superior. With the increase of plasma power, we get more ions generation which leads to more graphene growth. Plasma power exceeded beyond 225Watt; actually it was about 250 watt.

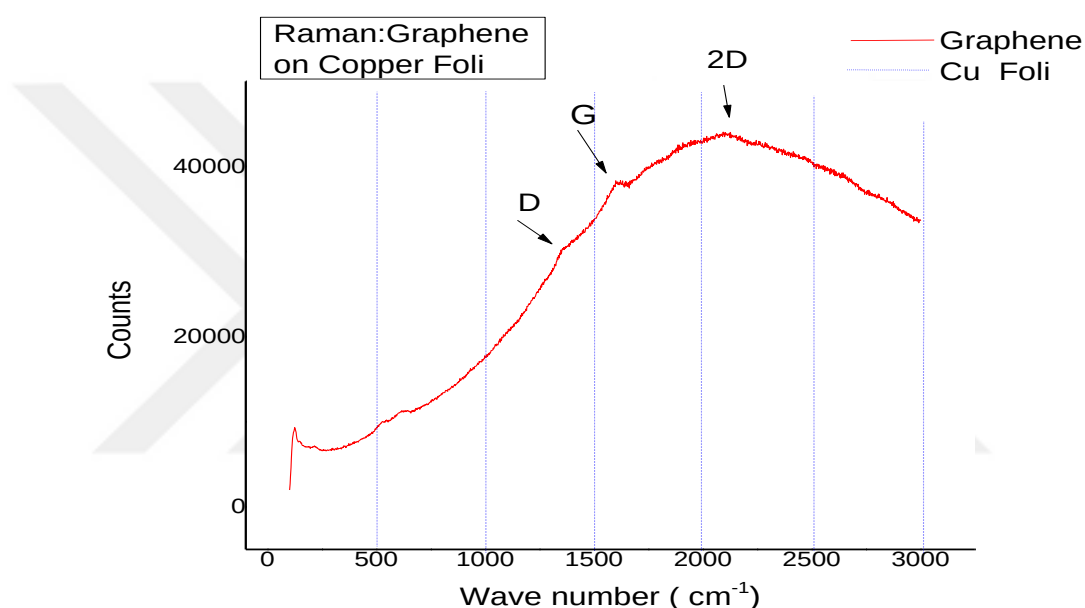


Figure 6.12. Raman spectra of PECVD graphene grown on Cu films at 750 °C

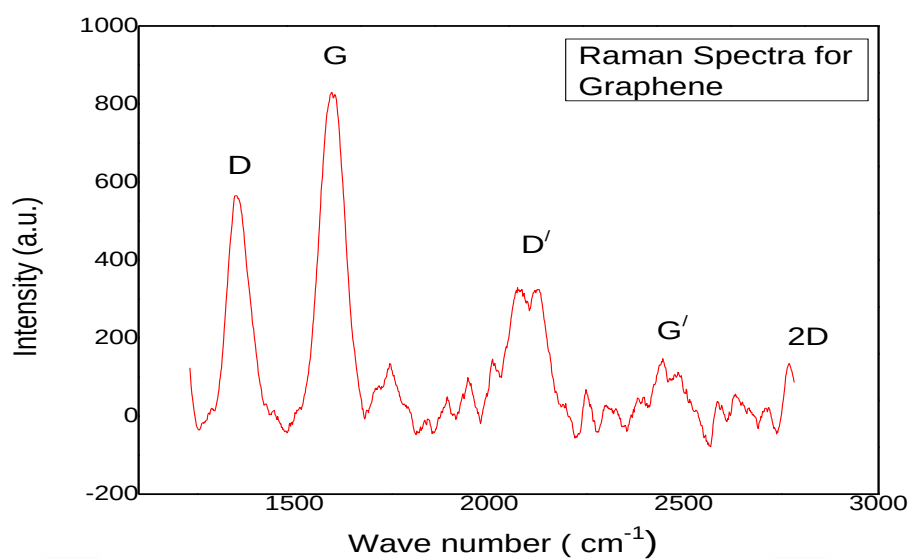


Figure 6.13. Raman spectra of single – walled carbon (graphene) synthesized at remote plasma power, at 850 C°, the insert shows D band, G band peak.

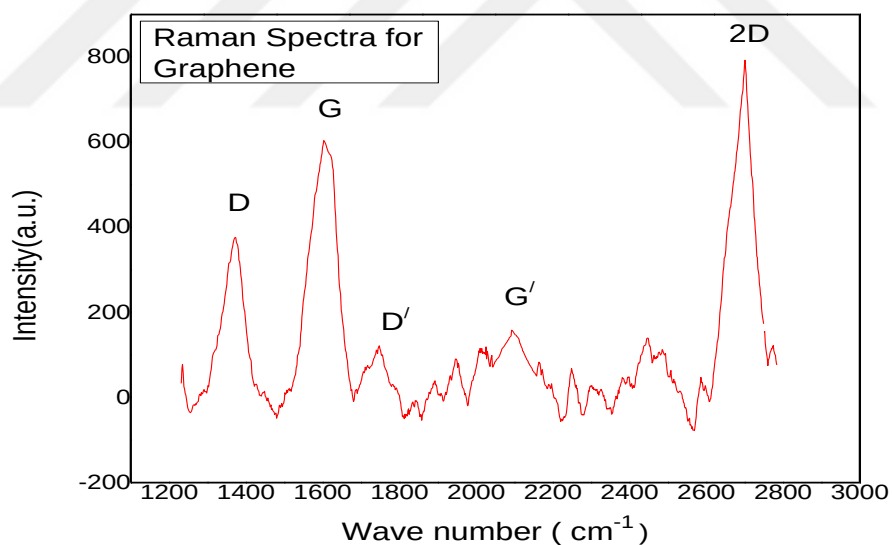


Figure 6. 14. Raman spectra of single – walled carbon (graphene) synthesized at remote plasma power, at 950 C° ,the insert shows D band ,G band peak.

We can put, o Figs. 6.13 and 6.14 in the same schema for the comparison of peaks as seen in Figure 6.15. In 2D (Two dimensions). Oslo we plot it three dimensions (3D) as we can see in Figure 6.16. For best compared between the two Figures.

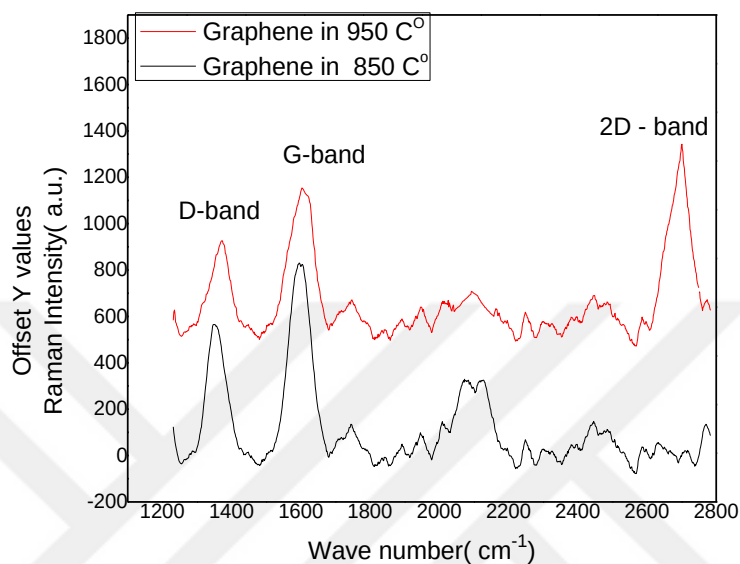


Figure 6.15. Raman spectra of single – walled carbon (graphene) synthesized at remote plasma power, the insert shows the correlation between plasma power and the D band ,G band peak and another's peaks at 850 C° ,950C° in (2D) two Dimensions.

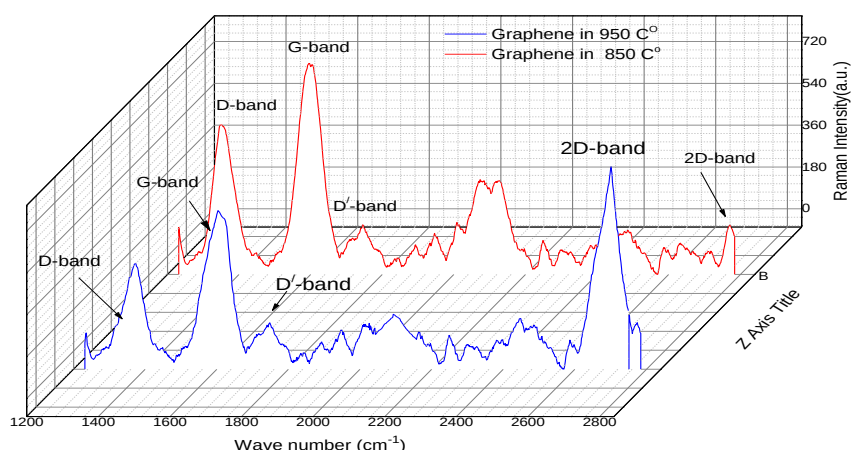


Figure 6.16. Raman spectra of single – walled carbon (graphene) synthesized at remote plasma power, the insert shows the correlation between plasma power and the D band, G band peak and another's peaks at 850 C° ,950C° in (3D) three Dimensions.

At first attempt, we grew graphene in 4 min and 750C°, in the second it was in 6 min and 850 C° , and in the the third it was in 8 min and 950 C°. Raman spectrum of graphene has three Main or dominant peaks, which denoted by D ,G and 2D at around 1342 cm⁻¹, 1598,7cm⁻¹ and 2110,5 cm⁻¹ ,respectively for the first one in 4 min as we can see in Table 1. We do the same for collecting the second and third samples as seen in Table 2 and Table 3. The ratio between the intensities of D band and G band peaks (which can be seen from the insert of Fig.1, 2, 3) was used to evaluate the purity of SWCNTs. The Raman D/G ratio at first decreased with an increase in plasma power up to 225W. After that, it increased when the plasma power was further increased to 250W. With the increase of plasma power, more ions were generated, which leads to more SWCNT growth. Therefore, these increases in graphene growth lead to the absorption purity index to increase, while for the intensities ratio for D/G decrease. We explained before that when the plasma power exceeds 200W, there will be a decrease in purity index and higher D/G ratio. There are two conditions for getting an increase in D/G peak area ratio: either by electrical or thermal reduction. In our experiment, we used thermal reduction method with

increase of plasma power. After that another method we tried to set plasma power was to raise the temperature.

In Raman spectroscopy, D band refers to the extent of defects while G band refers to the graphitic nature. Two points must be noted; the first is that if the D peak intensity is higher than G band, the sample has more defects in graphite network. These defects will appear imparted to the edges of the sheets. The second is that if the G band is prominent, sample can be said to be crystalline as graphene. The D and G band existence confirms the formation of graphene with lesser defects and crystalline structure. We can observe the piece in our graphs of the table 1, 2, 3.

Table 6. 8. Spectroscopic function's values for graphene, and the values calculating. Exp1. As illustrated in Fig.6.12

Sample	D	G	2D	$\frac{I_D}{I_G}$	$L_a = \frac{I_G}{I_D} C \lambda$	I(G)/(2D)
cm ⁻¹	1342,0	1598,7	2110,5	–	–	–
Intensity	30200	37923	43836	0,796350	5,52520	0,86511086

Table 6. 9. Spectroscopic function's values for graphene, and the values calculating. Exp2. As illustrated in Fig.6.13

Sample	D	G	2D	$\frac{I_D}{I_G}$	$L_a = \frac{I_G}{I_D} C \lambda$	I(G)/(2D)
cm ⁻¹	1345,4	1598,1	2768,6	–	–	–
Intensity	564,41	823,35	130,0	0,68550	6,41867	6,33346

Table 6. 10. Spectroscopic function's values for graphene, and the values calculating. Exp3. As illustrated in Fig.6.14

sample	D	G	2D	$\frac{I_D}{I_G}$	$L_a = \frac{I_G}{I_D} C \lambda$	I(G)/(2D)
cm ⁻¹	1368,6	1599,7	2699,4	–	–	–
Intensity	370,06	587,27	784,11	0,63013	6,9826	0,7489637

So we collected Raman spectroscopy data as (see Figure 6.16).

7. 1. CONCLUSION IN SUMMARY

There are many methods for observing graphene growth so we have assured that large-area graphene was grown on copper foils. We showed in our experiment that graphene synthesis on Cu foil surface by the PE-CVD process as a graphene coating. Continuous graphene film on Cu foil was grown using ethanol as a carbon feed-stock with a gas mixture of Ar (8.5 sccm). The Raman spectroscopy and SEM studies revealed few-layer graphene formation on Cu foil surface. Tarnishing of the Cu foil surface in presence of the ethanol vapor was investigated with and without coating the graphene film. It was found that the bare Cu foil surface immediately reacts with ethanol vapor to turn to the graphene-coated on Cu foil surface restrained reacting with the ethanol vapor, thereby preserving the Cu foil surface. Our findings showed that graphene film can be effectively synthesized on Cu foil substrate.

The synthesis method which presented in our paper may be expanded to the continuous production of large-area graphene-based films by using the array configuration of microwave plasma launchers and using another manufacturing method. To become a precursor for the manufacture of IC devices or manufacture of solar cells in the future.

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