

TUNABLE GRAPHENE PLASMONIC STRUCTURES WITH DIFFERENT GATING SCHEMES

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By
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We certify that we have read this thesis and that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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ABSTRACT

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The aim of this thesis is to examine graphene plasmonic structures which yields actively tunable spectral resonances and compare two different ways to gate graphene. Plasmonic structures that consist of periodic fractal gold squares on graphene are used to increase light-graphene interaction. We show by simulations and experiments that higher degree fractal structures result in higher spectral tunability of resonance wavelength. This is explained by more plasmonic localization of light for higher degree fractal structures. Furthermore, spectral tunability of a plasmonic structure integrated with graphene is investigated comparing two different schemes for electrostatic gating. The fabrication methods and fabrication steps of the devices with different gating schemes is explained in detail. Comparison of back-gating and top-gating schemes confirms that top-gating using ionic liquid is a more efficient gating method. Top-gating yields the same amount of spectral tunability while requiring smaller gate voltages compared to that of back-gating experiments.

Keywords: Optoelectronic devices, Graphene, Surface plasmons, Spectral tunability, Nano-fabrication, Electrical gating.

ÖZET

FARKLI GEÇİTLEME YÖNTEMLERİ İLE REZONANSI AKORT EDİLEBİLİR GRAFEN PLAZMONİK YAPILAR

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Bu tezin amacı akort edilebilir izgesel rezonans gösteren grafen plazmonik yapıları incelemek ve grafeni geçitlemek için iki farklı yöntemi karşılaştırmaktır. Grafen üzerinde periyodik altın fraktal karelerden oluşan bu plazmonik yapılar grafenin ışık ile etkileşimini artırmak amacıyla kullanılmıştır. Daha yüksek dereceli fraktal yapılarda daha fazla izgesel akort edilebilme olduğu simülasyonlarla ve deneylerle gösterilmiştir. Bunun sebebi yüksek dereceli fraktal yapılar ile daha fazla ışık lokalizasyonu sağlanmasıdır. Ayrıca, grafen plazmonik yapıların izgesel akort edilme özelliği iki farklı elektriksel geçitleme yöntemi ile incelenmiştir. Üstten geçitleme ve alttan geçitleme yöntemleri kullanılan iki aygıtın üretim methodları ve üretim adımları ayrıntılarıyla açıklanmıştır. Bu iki yöntemin karşılaştırılması sonucunda, iyonik bir elektrolit kullanılan üstten geçitleme yönteminin daha verimli olduğu belirlenmiştir. Üstten geçitleme ile alttan geçitlemeye nazaran çok daha düşük voltajlar uygulanarak aynı miktarda izgesel akort edilebilme gözlenmiştir.

Anahtar sözcükler: Optoelektronik aygıtlar, Grafen, Yüzey plazmonları, İzgesel akort edilebilme, Nano-üretim, Elektriksel geçitleme.

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Chapter 1

Introduction

In this thesis, we examine graphene plasmonic structures which yields actively tunable spectral resonances and compare two different ways to gate graphene. We aim to show that integrating graphene to a plasmonic system, such as periodic gold square fractals on SiO₂ dielectric, enables the spectral tunability of the plasmonic resonance. In addition to this, we investigate the relationship between increased plasmonic localization of light and the amount of spectral tuning. We demonstrate this by simulations and experiments of 1st, 2nd and 3rd degree square fractal structures. Lastly, we compare two different gating schemes, back and top gating, during the experiments.

The second chapter, Theoretical Background, includes the theoretical background needed to explain the phenomenon in the design, simulations and experiments. Electrical and optical properties of graphene is discussed in the first section. Unique band structure of graphene and details of its Fermi level dependent permittivity are studied. Following that, we explain how surface plasmons work. We explain the physics using the simple example of a metallic sphere in dielectric medium and understand the factors that affect surface plasmon resonances.

The design and simulations of the plasmonic structures are given in the third chapter, Plasmonic Square Fractal Structures. 1st, 2nd and 3rd degree square

fractal designs and why they are chosen are explained. Simulation setup of these structures with and without graphene as well as the discussion of the simulation results are included in this chapter.

The fourth chapter, Comparison of Two Different Gating Schemes, explains the experiments in detail. The design of the two different devices are introduced. Moreover, we explain the basics of the fabrication processes that we used. In the following sections, the fabrication steps of the first and second devices are described in detail. The last section of this chapter contains the experiment results.

The last chapter is the Conclusion. The simulation and experiment results are discussed in terms of the aims of the thesis. Suggestions for improvements are also mentioned.

Chapter 2

Theoretical Background

This chapter describes the theoretical background about graphene and plasmonics relevant to the following chapters of this thesis. Firstly, electrical and optical properties of graphene are introduced. After that, physics of surface plasmons is explained. The design and methods used in this thesis will be better understood in the light of this information.

2.1 Electrical and Optical Properties of Graphene

Graphene, is a 2D material which consists of a single layer of carbon atoms arranged in a honeycomb lattice (see Figure 2.1a). It has been a promising material for electro-optic devices due to its various remarkable properties. Graphene possesses carrier mobility values as high as $200\,000\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, which is the highest recorded value in literature so far [1, 2]. This ultra high mobility is a result of zero effective mass of traveling electrons [3] and electron mean free path values as high as $1\text{ }\mu\text{m}$ [4].

The unique band structure of graphene is illustrated in Figure 2.1b. Graphene is said to be a semi-metal, since the valence and conduction bands meet at the so called Dirac points. At the vicinity of the Dirac points valence and conduction

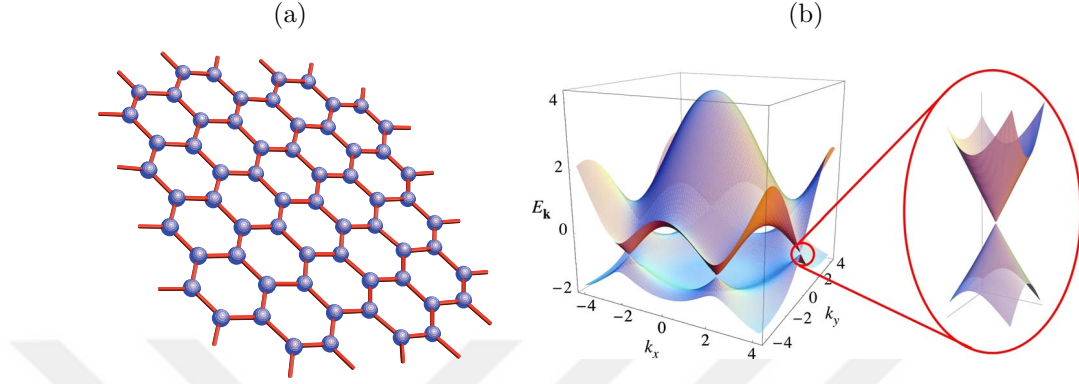


Figure 2.1: (a) 2D honeycomb lattice of graphene [5]. (b) Electronic dispersion in the honeycomb lattice [4].

bands have a conical shape, therefore the dispersion relation is linear. Near the Dirac point graphene has low density of states which allows meaningful changes in the Fermi level with variation of carrier concentration [6]. As a result of this and the confinement of electrons in one atomic layer, optical transitions in graphene can be effectively controlled by electrical gating.

The band structure of graphene and the allowed transitions for various cases are shown on Figure 2.2. For undoped graphene (see Figure 2.2a), Fermi level is at the meeting point of valence and conduction bands. Interband transitions at a wide range of frequencies are allowed. Furthermore, optical absorption is frequency independent and equal to 2.3% per layer, which is strong when the thickness of graphene is considered [7]. Figure 2.2b and Figure 2.2c show n-doped and p-doped cases respectively. In these cases, Fermi level is either at the valence or conduction bands, thus states with energies lower than the Fermi level are occupied. Thus, interband transitions to states with higher energy than the Fermi level are allowed. These interband transitions require a minimum energy of twice the Fermi level, ($\hbar\omega > 2E_F$). In Figure 2.2d, the case with intraband transitions is illustrated. In order for an intraband transition to take place scattering from phonons and defects is required for conservation of momentum [8]. Intraband transitions, therefore, are only relevant at frequencies where interband transitions can't satisfy the energy condition of $\hbar\omega > 2E_F$. As a result, optical conductivity of graphene is dominated by interband transitions at visible and

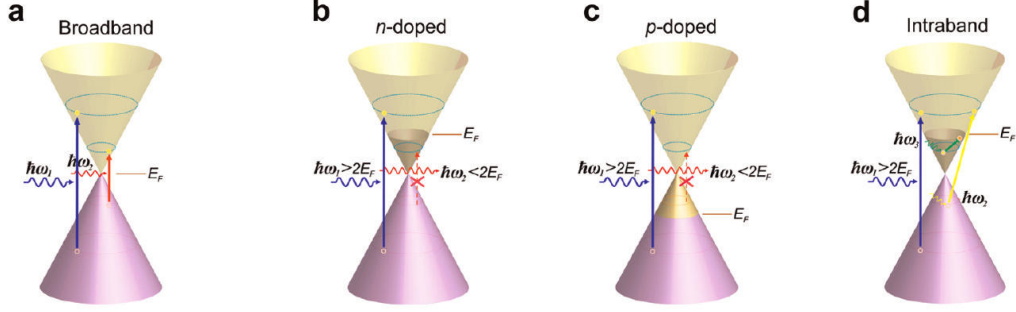


Figure 2.2: Interband and intraband transitions in the graphene band structure for different cases [7].

infrared frequencies; and by intraband transitions in the terahertz regime.

The optical conductivity of graphene can be modelled taking these interband and intraband contributions into account as given in Equation 2.1 [9, 10].

$$\begin{aligned} \sigma_s(\omega) = & \frac{2ie^2k_BT_L}{\pi\hbar^2(\omega + i/\tau)} \ln \left[2 \cosh \left(\frac{E_F}{2k_BT_L} \right) \right] \\ & + \frac{e^2}{4\hbar} \left[\frac{1}{2} + \frac{1}{\pi} \arctan \left(\frac{\hbar\omega - 2E_F}{2k_BT_L} \right) \right] \\ & - \frac{i}{2\pi} \ln \left(\frac{(\hbar\omega + 2E_F)^2}{(\hbar\omega - 2E_F)^2 + 4(k_BT_L)^2} \right) \end{aligned} \quad (2.1)$$

In this model, k_B is the Boltzmann constant, T_L is the lattice temperature, ω is the frequency, E_F is the Fermi energy, and τ is the carrier relaxation lifetime. Carrier relaxation lifetime, τ depends on σ , the semi-classical diffusive conductivity for 2D graphene, g_s and g_v , which are the spin and valley degeneracy factors respectively and are taken as 2 [11]. This dependence is given in Equation 2.2.

$$\tau = \sigma \frac{\hbar}{g_s g_v e^2} \frac{2\hbar}{E_F} \quad (2.2)$$

Finally, the permittivity of graphene is calculated from the conductivity using Equation 2.3.

$$\varepsilon(\omega) = \varepsilon_r + i\varepsilon_i = 1 + \frac{i\sigma_s}{\omega\varepsilon_0 t_G} \quad (2.3)$$

Both optical conductivity and permittivity of graphene are functions that have strong dependence on the Fermi level, E_F . The Fermi level of graphene can be

easily modified by controlling the sheet carrier concentration, n . The dependence of E_F on n is given in Equation 2.4 [3].

$$E_F = \hbar v_F \sqrt{\pi n} \quad (2.4)$$

Here, v_F is the Fermi velocity and is equal to $v_F = 10^6 \text{ ms}^{-1}$. These equations display the dependence of the graphene permittivity on the sheet carrier concentration. This relationship gives graphene its famous property of tunability. Electrical gating of graphene integrated with for example, a plasmonic system, allows for the spectral tunability of the resonances. Active control of spectral tunability of graphene plasmonic systems constitutes the backbone of the experiments in this thesis.

2.2 Localized Surface Plasmons

Metals are often modelled as fixed positive cores surrounded by a sea of free electrons. Hence, metals are sometimes referred to as plasmas, and the field that studies the oscillations of the electrons of this plasma is called plasmonics [12]. In this thesis, plasmonic systems will be used to increase light-graphene interaction via localized surface plasmons. In this section, we will explain how sub-wavelength sized periodic metallic particles can help localise fields into sub-wavelength volumes.

We can look at the simple case of a sub-wavelength metallic sphere to better understand the physics of localized surface plasmon resonances (see Figure 2.3). We assume that a homogeneous metallic sphere with a radius of a and dielectric function of $\varepsilon(\omega)$, is placed in a medium with a dielectric constant of ε_m . An electric field, E_0 , is applied. This electric field will cause the free electrons of the sphere to move in the opposite direction of the field, leaving behind static positive cores. Hence, a dipole moment $\mathbf{p}$ will be induced inside the sphere. The relationship between the dipole moment and the applied field is explained via Equation 2.5.

$$\mathbf{p} = \varepsilon_0 \varepsilon_m \alpha \mathbf{E}_0 \quad (2.5)$$

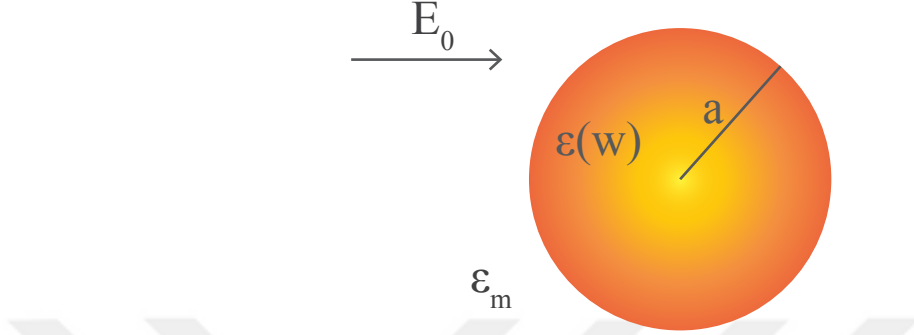


Figure 2.3: Schematic of a sub-wavelength metallic sphere placed in a dielectric medium with an applied E-field.

Here, α is defined as the polarizability of the sphere. Under the electrostatic approximation the polarizability can be calculated using Equation 2.6 [13].

$$\alpha = 4\pi a^3 \frac{\varepsilon - \varepsilon_m}{\varepsilon + 2\varepsilon_m} \quad (2.6)$$

Note that, polarizability depends on the size of the particle, dielectric function of the metal and the dielectric constant of the surrounding medium. Equation 2.6 also reveals the resonant behaviour of polarizability. When the term $|\varepsilon + 2\varepsilon_m|$ is minimum, a resonant enhancement of polarizability occurs. This resonance is referred to as the dipole surface plasmon resonance. This resonance occurs when the frequency of the incident field matches the natural frequency of the free electrons oscillating against this field. In Figure 2.4, the magnitude and phase of polarizability of a silver particle in air medium with respect to the energy of the applied field are shown. $\varepsilon(\omega)$ is taken as the Drude model of silver. Here, resonant behaviour is observed when the condition $\varepsilon(\omega) = -2\varepsilon_m$ is satisfied.

As a direct consequence of the resonant polarizability, the scattering and absorption behaviour of the sphere is also resonant and enhanced greatly at the dipole surface plasmon resonance. When this metallic sphere is illuminated with a plane wave, scattering cross section, C_{sca} , and absorption cross section,

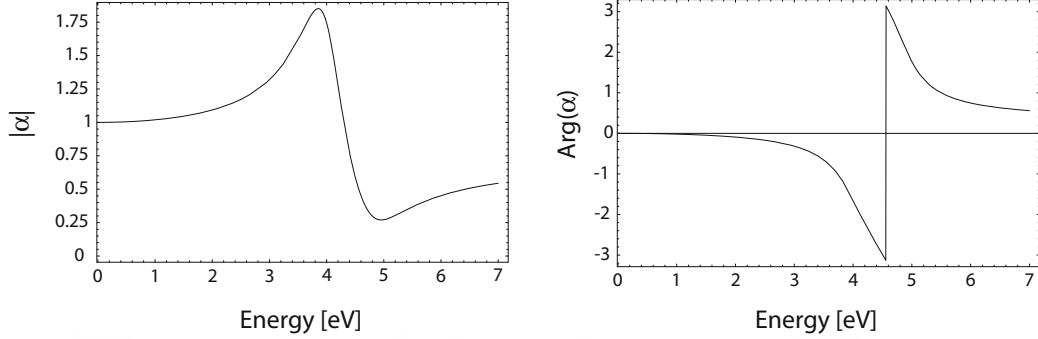


Figure 2.4: The magnitude and phase of polarizability of a silver particle in air medium with respect to the energy of the applied field. [13].

C_{abs} , are in the following form [13].

$$C_{sca} = \frac{k^4}{6\pi} |\alpha|^2 = \frac{8\pi}{3} k^4 a^6 \left| \frac{\varepsilon - \varepsilon_m}{\varepsilon + 2\varepsilon_m} \right|^2 \quad (2.7)$$

$$C_{abs} = k \text{Im}(\alpha) = 4\pi k a^3 \left(\frac{\varepsilon - \varepsilon_m}{\varepsilon + 2\varepsilon_m} \right) \quad (2.8)$$

Equation 2.6 and 2.7 show that scattering and absorption cross sections have a^6 and a^3 dependence respectively. For sub-wavelength spheres with a radius much smaller than the wavelength, the efficiency of absorption dominates that of scattering. Extinction cross section, C_{ext} , is defined as the sum of absorption and scattering cross sections. Figure 2.5 illustrates the resonant behaviour of the extinction cross section of a silver spheres in two different dielectric media.

So far we considered the case of a single metallic sphere excited by an electric field. The physics behind this special case is helpful in understanding the surface plasmon resonances. Of course, metallic sub-wavelength sized particles of any shape can involve surface plasmon resonances. Figure 2.6 is from a study that shows the effect of size and shape on the resonance frequency. The scanning electron microscopy (SEM) images of the metallic nanoparticles and their dark-field (DF) images are shown. Different shapes of materials show scattering resonances at different visible frequencies.

In the case of multiple metallic particles arranged periodically, dipole surface

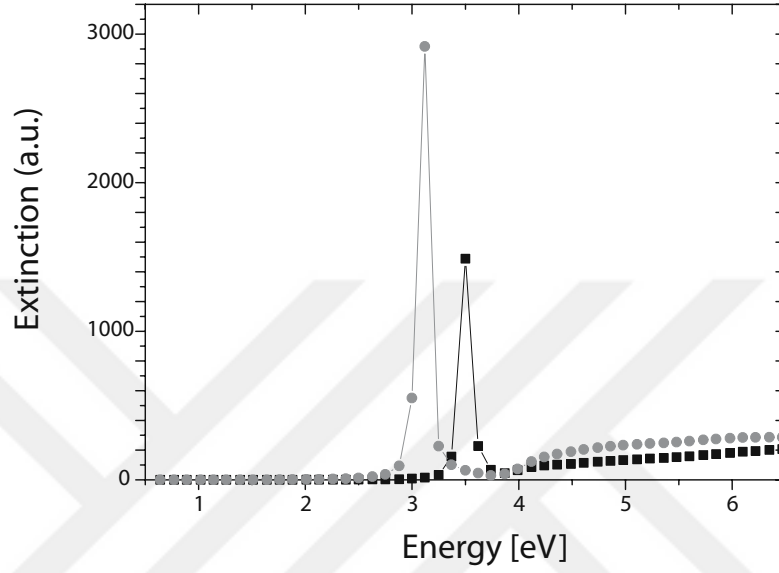


Figure 2.5: Extinction cross section of a silver sphere in air (black) and silica (gray) media [13].

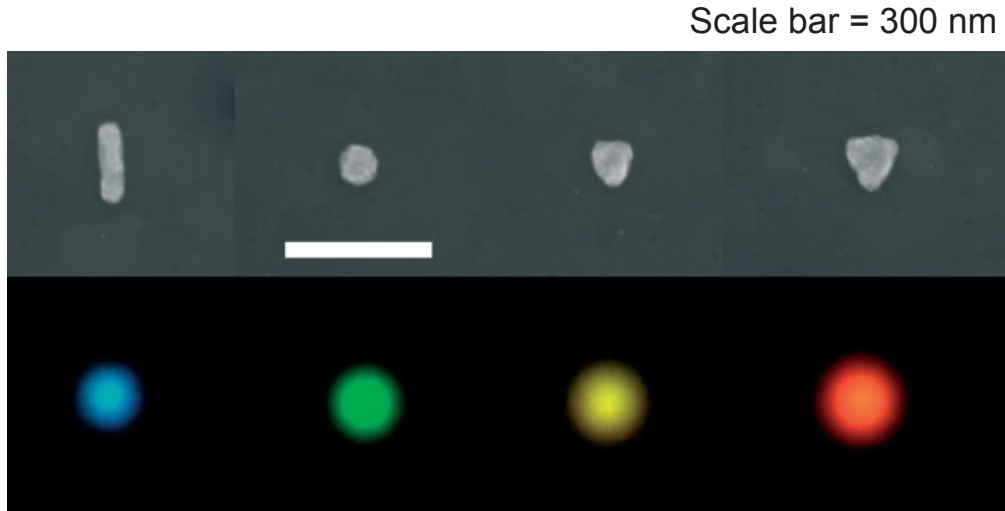


Figure 2.6: The scanning electron microscopy (SEM) and dark-field (DF) images of metallic nanoparticles with various shapes [12].

plasmons couple if the metallic particles are closely spaced enough to interact with each other's modes. The coupling between adjacent dipole surface plasmons, forms the strong non-propagating localized fields that are referred to as the localized surface plasmons. These systems are beneficial because of their ability to concentrate fields in small volumes. The wide range of areas for their applications include [14] ultrasensitive detection [15], photovoltaics [16], nanoscale photometry [17], cancer therapy [18], and nonlinear optics [19]. In this thesis, we will be using periodic sub-wavelength metallic structures in order to increase light-graphene interaction by the help of localized surface plasmons.

Chapter 3

Plasmonic Square Fractal Structures

3.1 Design of the Plasmonic Structures

In the previous chapter, we mentioned that the absorption of undoped graphene has a value equal to 2.3% per layer for a broad frequency range [7]. This absorption is high considering that the material consists of only one atomic layer, however it is not sufficient for most photonic applications. As explained earlier, we can use plasmonic structures in order to localise the field around graphene and subsequently increase graphene-light interaction.

Gold plasmonic structures on graphene that lays on a SiO_2 substrate can serve this purpose. Localised surface plasmon resonances can be tuned by controlling the sheet carrier concentration of graphene. Gold square patterns of different fractal degrees are designed in order to compare the effects of plasmonic interaction on tunability. We expect that higher degree fractal structures will enable higher spectral tunability.

The geometry of 1st, 2nd and 3rd degree square fractal unit cells are shown in

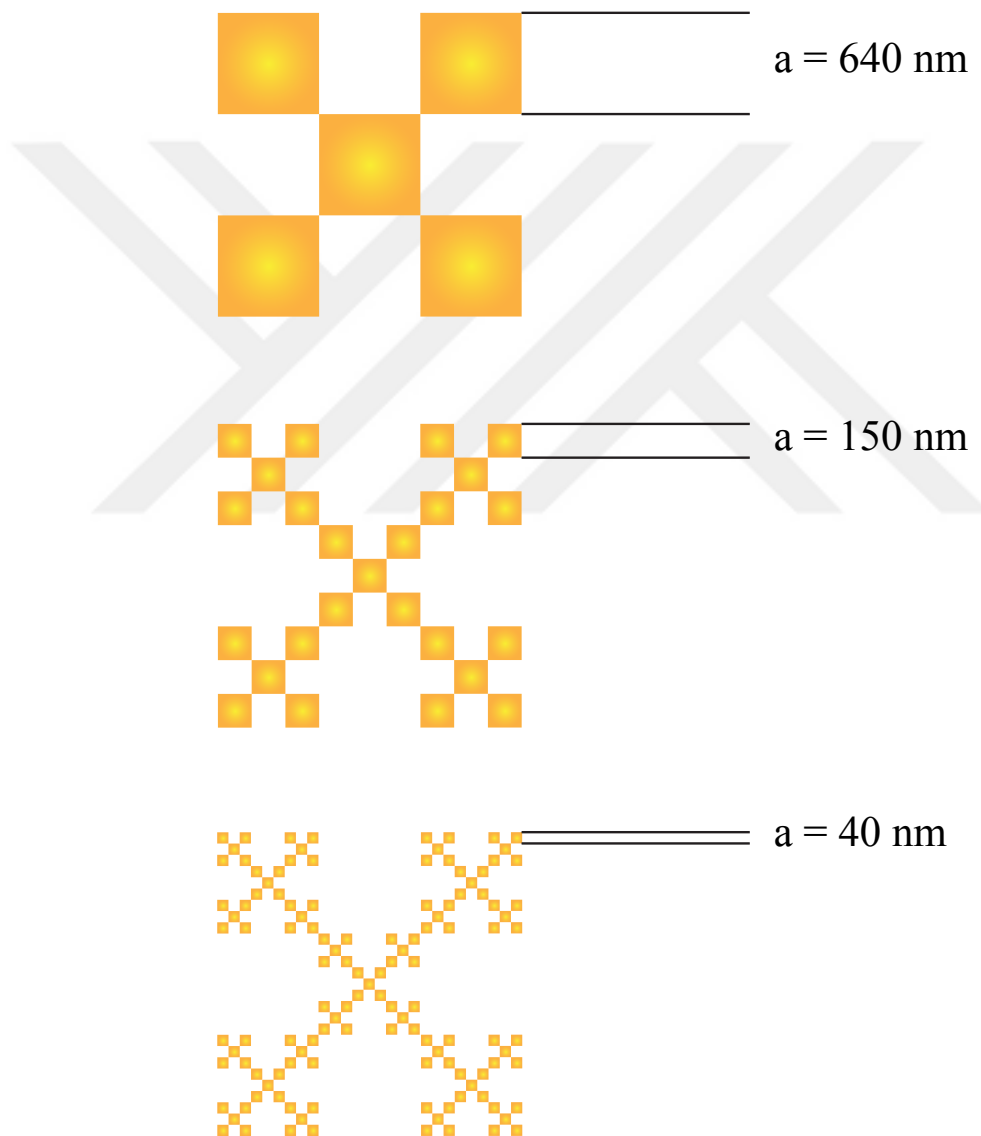


Figure 3.1: 1st, 2nd and 3rd degree square fractal unit cells.

Figure 3.1. The periodicity, p , and the side length of the smallest square unit, a , are chosen to achieve resonance peaks at similar wavelengths (around 6 - 6.5 μm). (p, a) values of 1st, 2nd and 3rd degree fractals are (2400,640), (1800,150) and (1600,40) nm respectively.

3.2 Numerical Simulations

The numerical simulations are carried out by using the finite-difference time domain simulation software, Lumerical FDTD Solutions. In the simulation setup, 280 nm thick SiO₂ is layered on an infinite silicon slab. On top of that 50 nm thick gold squares are placed to form different degree fractal unit cells. Periodic boundary conditions are used on the sides of the unit cell while using PML (Perfect Matched Layer) boundary conditions in the normal direction. The structure is illuminated with a normally incident plane wave source and a reflection monitor was placed above the source. The 3D view and side view of the simulation setup is given in Figure 3.2a-b.

Firstly, a set of simulations without graphene is carried out in order to compare the electric field localisations for different fractal patterns. Figure 3.3 illustrates the electric field distribution taken from a monitor that lays on the SiO₂/gold interface where graphene would lay. For each fractal degree, the electric field distribution at the reflection resonance wavelength are illustrated. The electric field distribution shows that the intensity of the localised field and the number of localisation centres both increase going from 1st to 3rd degree fractals. Hence, light-graphene interaction is greater for higher degree fractal geometries.

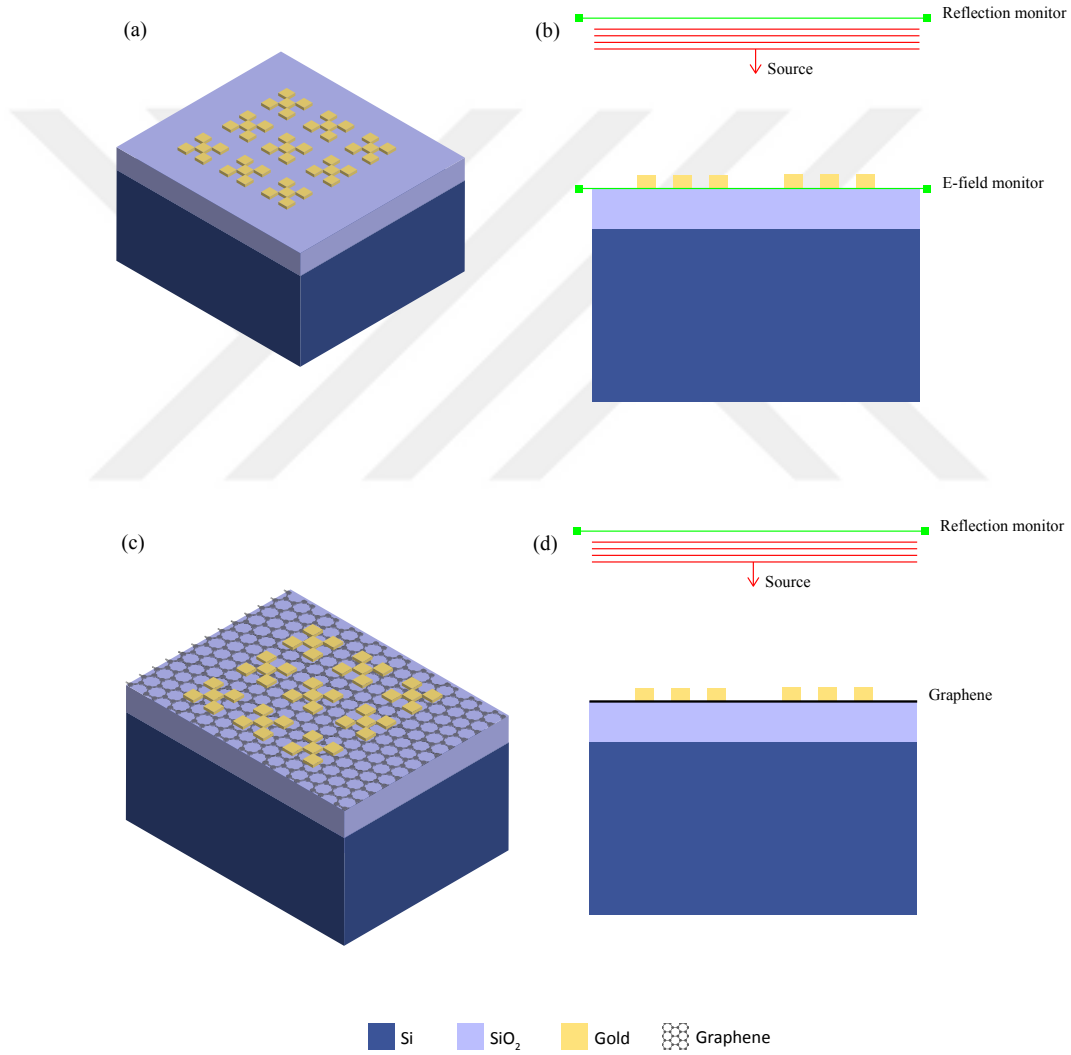


Figure 3.2: The (a) isometric view and (b) side view of the simulation setup without graphene. The (a) isometric view and (b) side view of the simulation setup with graphene.

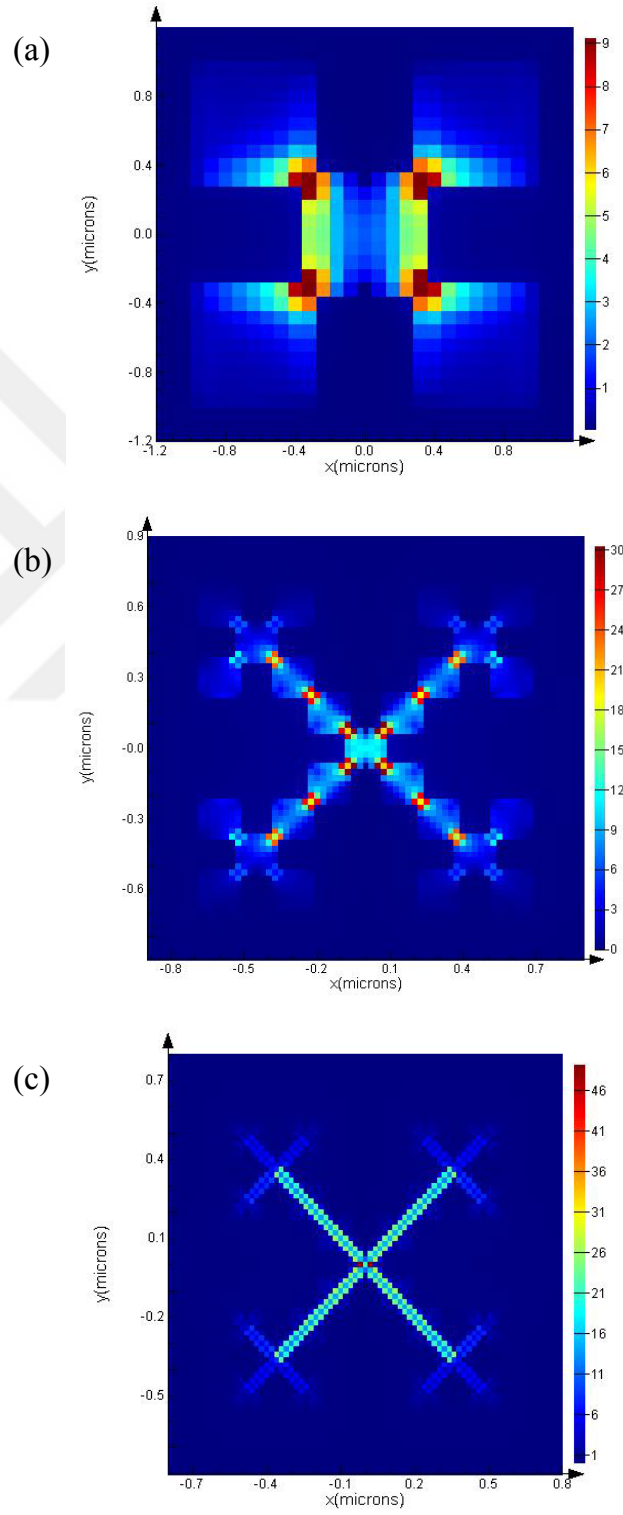


Figure 3.3: The electric field localisations at the reflection resonance wavelength for (a) 1st, (b) 2nd and (c) 3rd degree fractal patterns.

To observe the effect of increased electric field localisation on the spectral tunability we have done simulations with graphene. The simulation setup with graphene is illustrated in Figure 3.2c-d. A 1 nm thick graphene material is added between the SiO₂ slab and the gold patterns. The permittivity of this graphene material is modelled according to Equations 2.1-3. The permittivity plots of gold and graphene with different Fermi levels are shown in Figure 3.4. Two sets of simulations with different graphene Fermi levels are carried out for each of the 1st, 2nd and 3rd degree structures. The Fermi level value of the graphene model is taken as 0.2 eV for one set of simulations and 0.7 eV for the other. The reflection spectra of the simulations are shown in Figure 3.5. According to the simulation results, the reflection resonance peak can be tuned by changing the Fermi level of graphene. This is explained by the Fermi level dependence of graphene permittivity (see Equation 2.1). Furthermore, the spectral tunability increases as the degree of fractal patterns increases. This is an expected result since we have seen that electric field localisation is greater for higher degree fractals. The increased light-graphene interaction results in higher tunability.

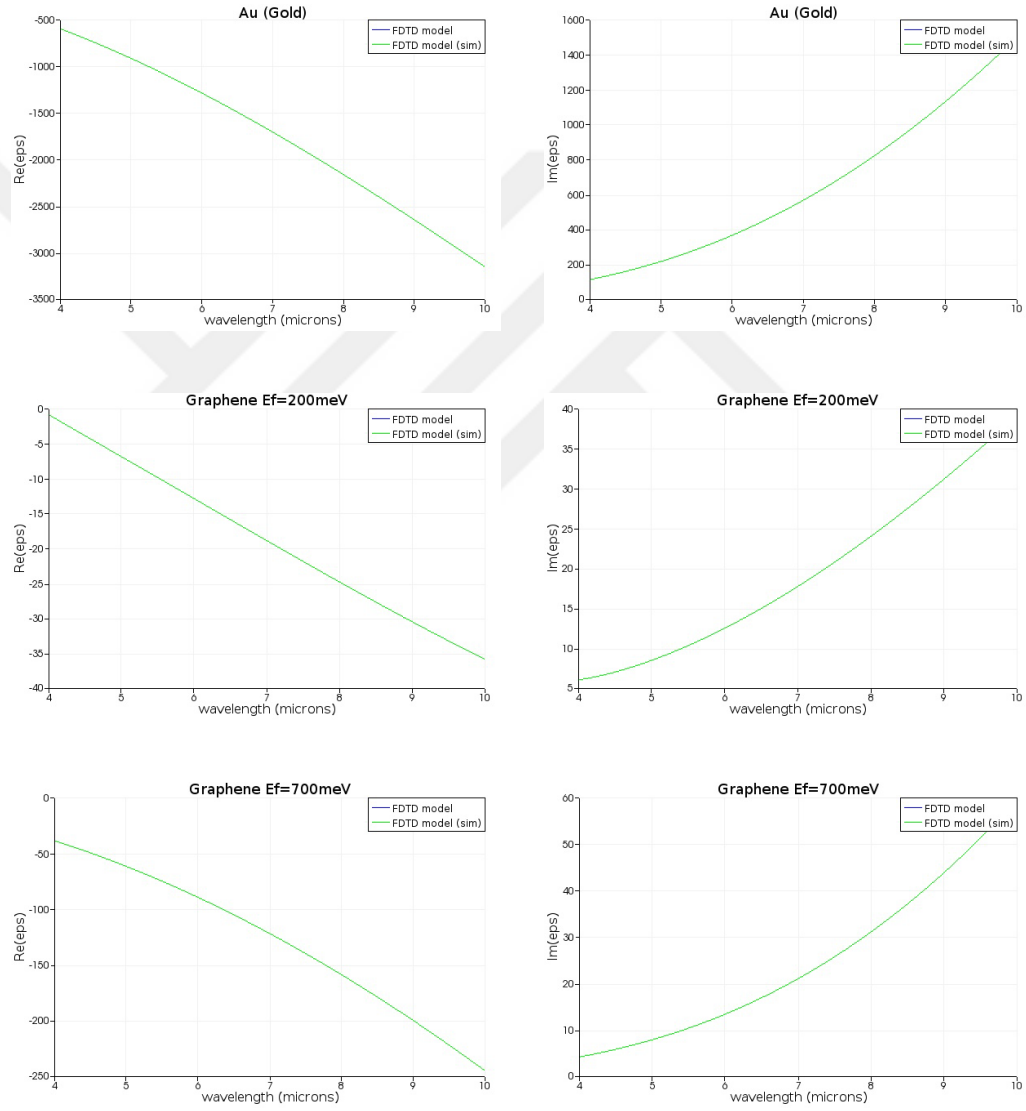


Figure 3.4: The real and imaginary parts of permittivity o gold, graphene with 0.2eV and 0.7 eV Fermi levels.

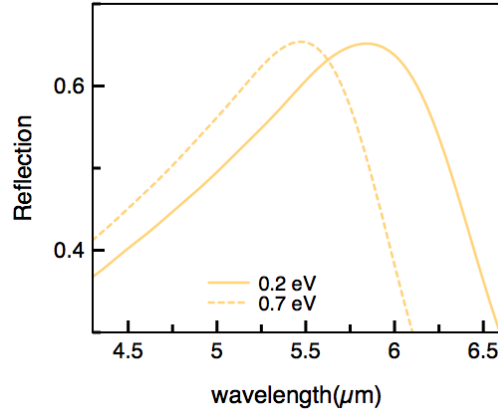
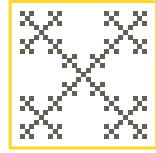
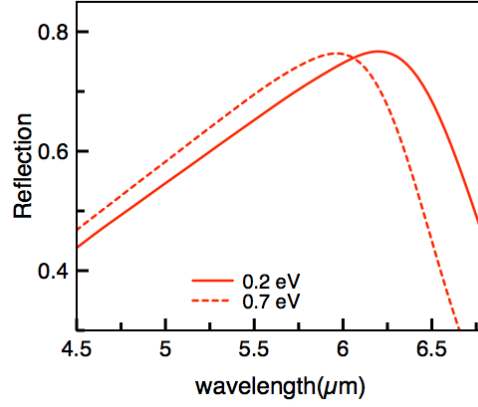
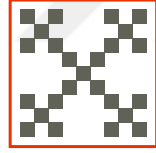
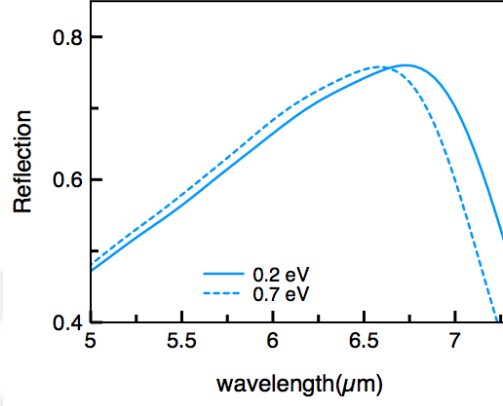
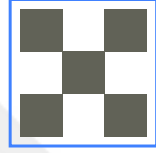


Figure 3.5: The simulation results of the spectral tunability of 1st (blue), 2nd (red) and 3rd (yellow) degree fractal patterns. In each graph, solid lines and dashed lines represent simulations with Fermi level values of 0.2 and 0.7 eV, respectively.

Chapter 4

Comparison of Two Different Gating Schemes

4.1 Proposed Gating Schemes

We carried out experiments of the designed structures to verify the simulation results. In order to control the sheet carrier concentration of graphene, traditional back gating on a highly doped Si/SiO₂ substrate is a reliable method to manipulate its carrier concentration. This approach may require control voltages as high as 390 V for operation [20]. Back gated graphene devices have been used in many applications as photodetectors [21], modulators [20, 22], and nanoresonators [23]. On the other hand, a more efficient dielectric, the ion gel has been used by Halas et al. [24] and Ju et al. [25] to top gate a patterned graphene layer with much lower control voltages.

We compare these two gating schemes by fabricating two separate devices and conducting experiments. Carrier density of graphene is manipulated by using back gating and SiO₂ dielectric for the first device (see Figure 4.1a). The second device is top gated using ionic liquid as a conducting medium(see Figure 4.1b).

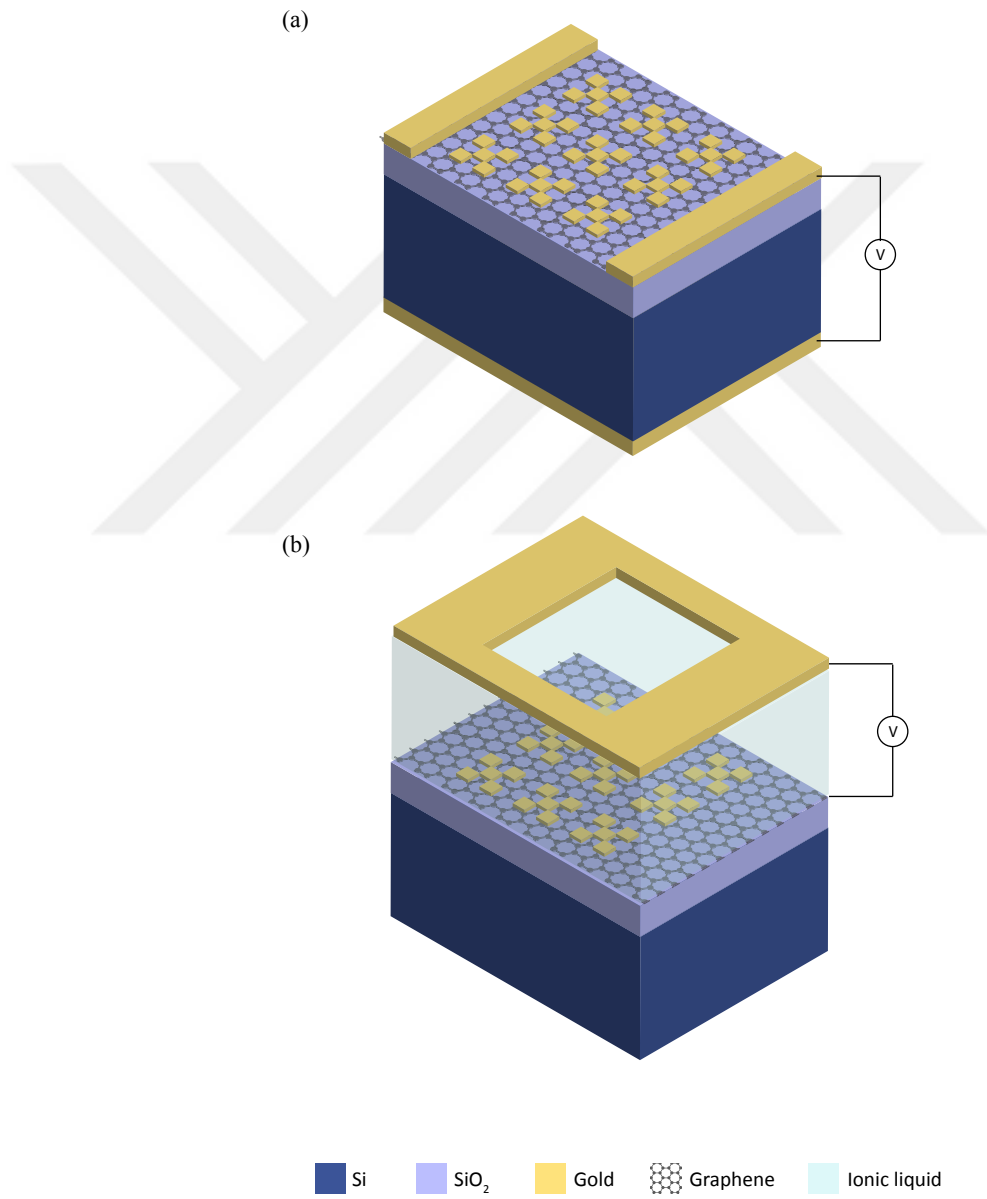


Figure 4.1: Isometric view of the measured (a) back gated and (b) top gated devices.

4.2 Fabrication Process Basics

4.2.1 Photolithography

Photolithography is a popular micro-fabrication technique which provides high throughput and minimum feature sizes around 2-3 μm [26]. To prepare the samples, firstly are covered with a photosensitive material, called a photoresist. A photoresists' solubility is altered when exposed to specific wavelengths of light. The basic principle of photolithography is to illuminate some areas of the resist with ultra-violet light and in turn be able to remove either the exposed or un-exposed parts in a specific solvent. A photomask, which has opaque patterns on a transparent substrate, is used while exposing the resist in order to control the areas where light falls. The alignment between the sample and the mask, as well as the exposure after the alignment is done via a mask aligner. The factors that effect the feature sizes include photoresist thickness, exposure time, proximity/contact mode and develop time.

In our laboratories, we are using the MA6 system by the SÜSS MicroTec company. The system provides proximity, soft contact, hard contact, and vacuum contact lithography modes. While choosing between these modes, there is a trade off between resolution and contaminating the photomask. The system has a Hg lamp source and selective optical filters. The wavelength of output light is 365 nm which is referred to as the i-line. It is important to choose photoresists which are sensitive to the wavelength of operation.

The fabrication steps of our devices (see Figure 4.1) involves two different photolithography techniques: positive and image reversal photolithography.

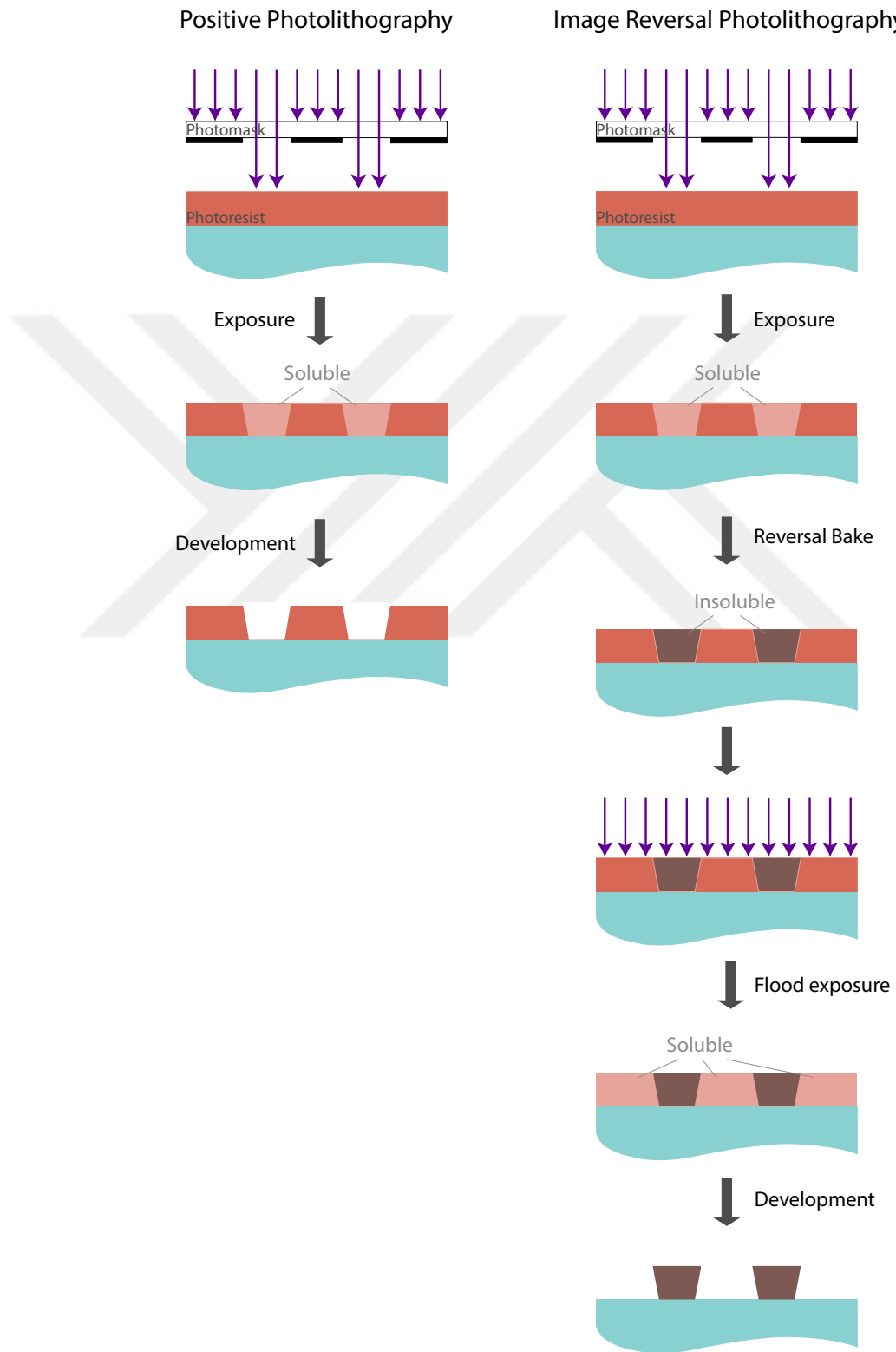


Figure 4.2: Positive and image reversal photolithography process steps.

4.2.1.1 Positive Photolithography

Positive photolithography is the process where the patterns on the mask are directly transferred onto the resist. Figure 4.2 illustrates the steps of positive photolithography. Positive photolithography only involves one exposure step which is realized after the photomask is carefully aligned to the sample. During this exposure, parts of the photoresist that was exposed to light becomes soluble. The special type of solvent that only removes exposed parts of the resist is called a developer. After development, only the parts not exposed to ultra-violet light will remain on the sample. Therefore, this pattern is the positive image of the opaque patterns on the photomask.

4.2.1.2 Image Reversal Photolithography

At the end of image reversal photolithography, photoresist patterns is a reversed image of photomask patterns. The steps of image reversal photolithography are also illustrated in Figure 4.2. The initial steps of the image reversal photolithography is similar to positive photolithography: the sample under the photomask is exposed to ultra-violet light and exposed parts of the photoresist becomes soluble. The critical step is the image reversal bake, where the sample is baked on a hotplate. This bake crosslinks the exposed parts, causing them to become insoluble, whereas unexposed parts remain photoactive. Flood exposure that requires no photomask, exposes all the resist. Therefore, the previously unexposed parts become soluble after flood exposure. However the developer is unable to solve the crosslinked parts. As a result, after the development the photomask pattern is reversed.

4.2.2 Electron Beam Lithography

Electron beam lithography is an advanced nano fabrication technique that is preferred when ultra high resolution is required. The basic principle is to scan an

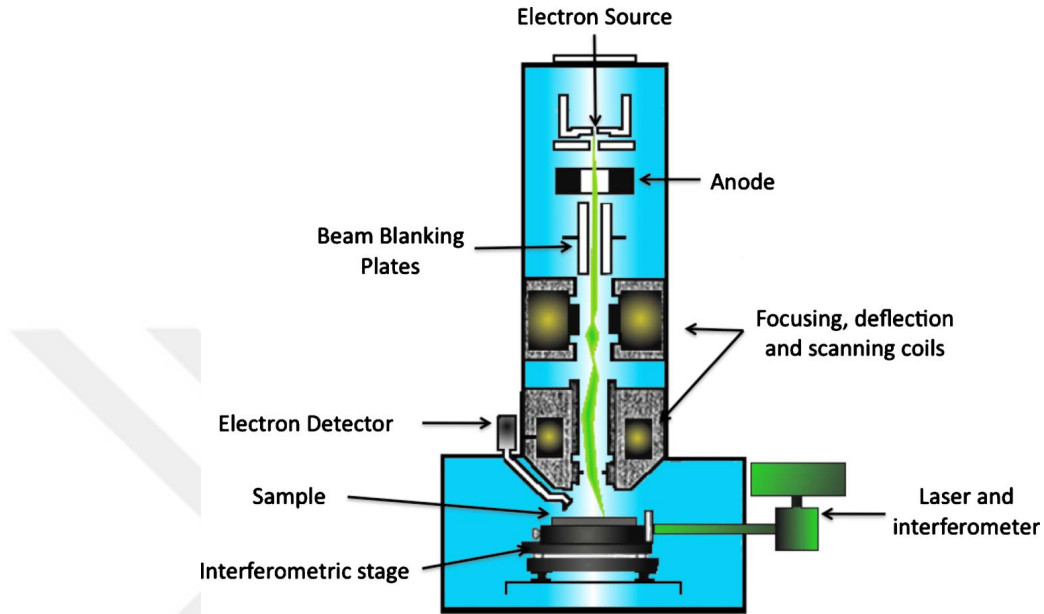


Figure 4.3: Schematic of an electron beam lithography system[27].

electron beam on the surface of an electron beam resist along an arbitrary path and pattern the electron beam resist accordingly. Electron beam's ultra short wavelengths in the order of a few nanometers [26] provide resolutions below 5 nm[28]. The main disadvantage of electron beam lithography is its low throughput. Resist thickness, exposure dose, develop time, and electron beam spot size are amongst the factors that effect resolution[29]. In order to achieve the smallest possible electron beam spot size, focus and stigmation settings should be adjusted accurately.

Figure 4.3 shows the schematic of a typical electron beam lithography system. The electron beam is first accelerated in the anode. The extra high tension (EHT) of the accelerator is set by the user depending on the application. While choosing EHT the trade-off between forward scattering (scattering of the beam when it enters the resist) and backward scattering (scattering of the beam when it reflects from the substrate) should be considered. Aperture size also affects the beam spot size significantly and is chosen depending on the desired feature sizes.

The process starts with spin coating an electron beam resist similar to photolithography. After spin coating and baking the resist it is important to coat the sample with a conductive polymer like aquaSAVE. This conductive polymer helps reduce the scattering of electrons inside the resist and improves resolution. There are no physical masks in electron beam lithography. The patterns are drawn through the software of the system and the beam is controlled by the software in a path to produce the desired patterns. The writefield, focus and stigmation of the beam, step size, exposure dose, aperture size and EHT values are set before each session. After electron beam exposure, the conductive polymer is removed and the sample is developed with a special developer that works with the electron beam resist of choice. The end result is positive or negative image of the mask depending on the type of the resist. In our laboratories we are using the eLine system by Raith GmbH.

4.2.3 Electron Beam Evaporation

Electron beam evaporation, is a type of physical vapor deposition. In this process, the source material is heated and eventually evaporated by focusing an electron beam on it. The process takes place in a vacuum chamber, where wafer holders are positioned on top of the source material. Schematic of an electron beam evaporator system is shown in Figure 4.4. This system is suitable for materials with high evaporation temperatures because by electron beam heating target materials can easily go up to high temperatures[30]. Evaporation rate can be adjusted by setting the current and accelerator voltage of the electron beam. The vapor of the material follows a linear path to the sample, therefore minimum amount of deposition occurs on the resist side walls. This provides ease of lifting the resist off after evaporation.

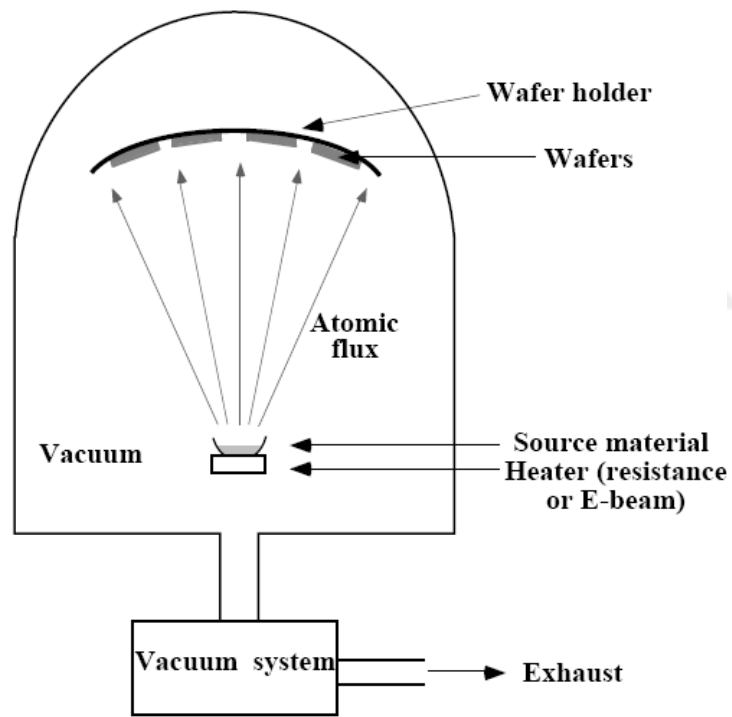


Figure 4.4: Schematic of an electron beam evaporator system[30].

4.2.4 Dry Etching

The last fabrication method that we will discuss is dry etching, namely ICP-RIE etching. ICP-RIE etching is one of the most vigorous etching techniques because both chemical and physical mechanisms contribute to etching. The plasma in ICP-RIE systems contain both free radicals and ions. ICP stands for inductively coupled plasma, which is generated by an RF source. The plasma contains free radicals which react with the sample to chemically etch it away. Chemical etching is therefore an isotropic process. During reactive ion etching (RIE), on the other hand, the ions that the plasma sustain are accelerated towards the sample via an applied bias. Physical etching occurs when the accelerated ions hit the sample's surface and drill through the sample. Thus, this process is an anisotropic process.

In our laboratories, we are using SAMCO ICP-RIE system. We prepare etching recipes where we specify types and flow rates of gasses, pressure inside the chamber, RF power, bias power and process duration. The pressure and gas flow rates are crucial for the formation of plasma. The RF and bias power determines how much physical and chemical etching contributes, therefore effects the directionality.

4.3 Fabrication of the Back Gated Device

The back gated device has three contacts; source and drain contacts on graphene and a back contact taken from under the p-doped silicon substrate (see Figure 4.1a). Fractal patterns are on the graphene patch that is between the source and drain contacts. We started fabrication with 10 mm $\times$ 10 mm CVD grown monolayer graphene on Si/SiO₂ samples purchased from Graphene Supermarket. 8 devices were fabricated from each sample. Each fabrication step is explained in detail in the following subsections.

4.3.1 Photomask Preparation

Firstly, we prepared the photomask to be used in the photolithography processes later on. We started with a quartz substrate and cleaned the sample with acetone and isopropyl alcohol (IPA). The patterns on the mask were fabricated by electron beam lithography. We used polymethyl methacrylate (PMMA) A6 which is a positive electron beam resist. We spin coated PMMA A6 at 2000 rpm for 40 seconds and baked on a hot plate at 180°C for 90 seconds. We choose a high aperture size of $60\mu m$ and set EHT to 10 kV and applied an area dose of $120\mu C/cm^2$. After the electron beam exposure, we developed the sample in a 1:1 MIBK (methyl isobutyl ketone) : IPA solution for 40 seconds. To stop the development sample is then put into IPA. Finally, we deposited 120 nm thick chromium via electron beam evaporation and lifted the excess off. The finished photomask is given in Figure 4.5. The patterns on the left are for graphene patches and the ones on the right are for source-drain contacts.

4.3.2 Patterning Graphene

In this step we formed isolated graphene islands for the active region of each device. We spin coated Si/SiO₂/graphene sample with TI35ES photoresist at 4000 rpm at 40 seconds. We removed the edge bead of the resist carefully using a Q-tip swab which is doused in acetone. We then baked the sample on hot plate at 110°C for 2 minutes. We did a positive lithography process, since we wanted the resist pattern to be the same as the mask pattern. Mask aligner was operated in hard contact mode and the left part of the mask shown in Figure 4.5 was used. After properly aligning the sample under the mask, we exposed the sample for 20 seconds at 10 W lamp power. After exposure, we developed the sample in 1:4 AZ400K : DI water for 25 seconds and development was stopped in DI water. After development, we had rectangular shaped resist islands on the sample and it was ready for O₂ plasma etching via ICP-RIE system. We used a recipe with bias power and ICP of 20 W and 50 W respectively, for 30 seconds. During this process, the graphene which was covered with resist was protected, however the

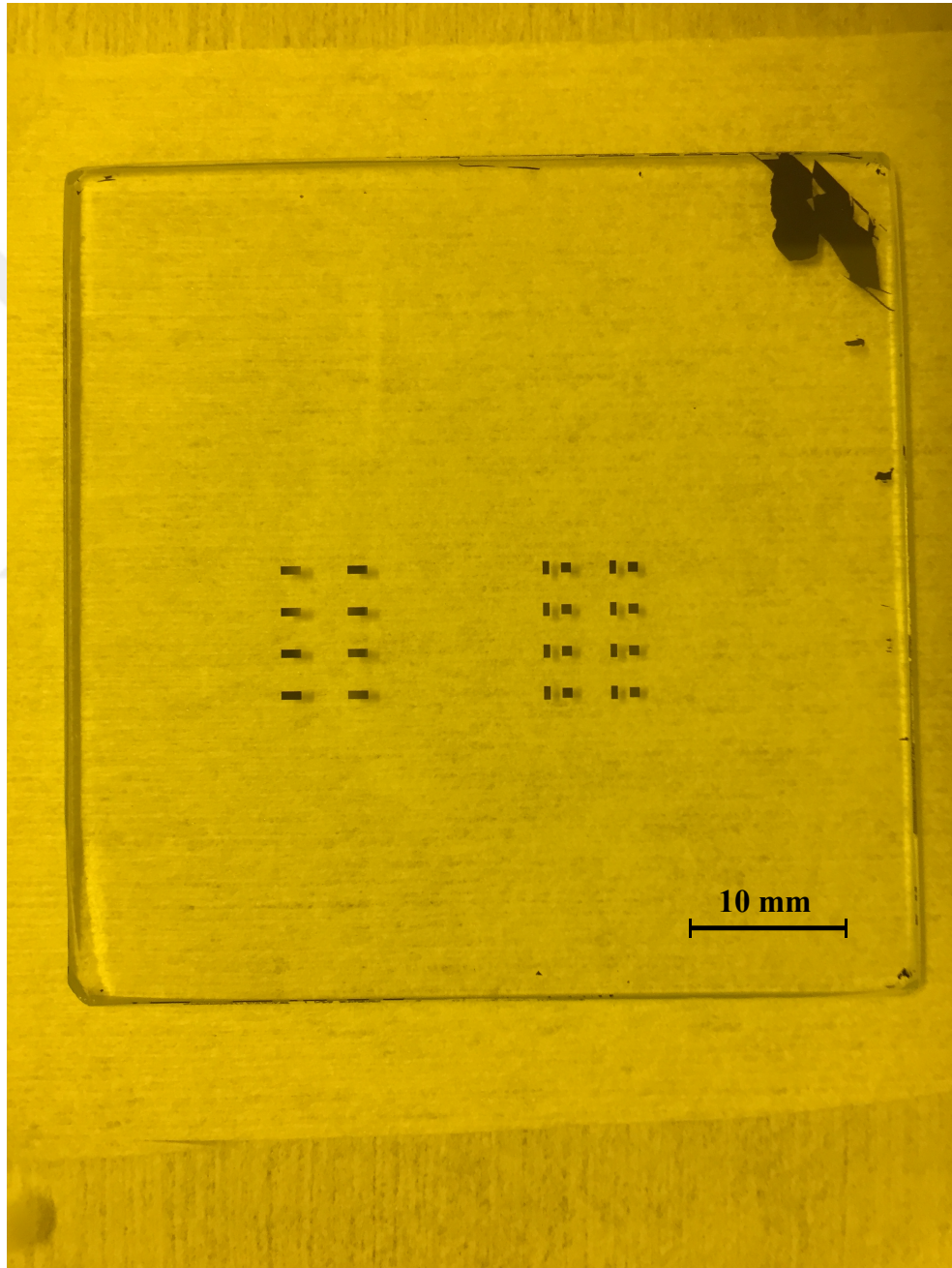


Figure 4.5: Photo of the photomask prepared for the lithography processes.

rest of the graphene was etched away. After cleaning the resist with acetone, we were left with 8 rectangular shaped graphene patches on the sample.

4.3.3 Fabrication of Source and Drain Contacts

To fabricate source and drain contacts we did an image reversal photolithography. Using the same photoresist of the previous process, we spin coated the sample with TI35ES photoresist at 4000 rpm at 40 seconds. We removed the resist edge bead and baked the sample at 110°C for 2 minutes on hot plate. Mask aligner was used in hard contact mode but this time the right part of the mask shown in Figure 4.5 was used. We did the alignment making sure that both contacts was overlapping with the previously isolated graphene regions. First exposure lasted 20 seconds and after that reversal bake was done at 120°C for 2 minutes on hot plate. Then, the photomask was removed and a flood exposure of 31.3 seconds was done. We finally developed the sample in 1:4 AZ400K : DI water for 20 seconds. At this point, the resist pattern was a negative image of the mask pattern. We evaporated 50/400 nm Ti/Au on the sample and lifted-off the photoresist. What was left was the source and drain contacts on the sides of the graphene patch. Figure 4.6 illustrates a microscope image of the device.

4.3.4 Fabrication of Square Fractal Patterns

The fractal patterns we designed contain feature sizes as small as 40 nm therefore we need to use electron beam lithography system for their fabrication. We decided to make $100\ \mu m \times 100\ \mu m$ regions of periodic fractal structures and prepared the masks on the eLine software. To prepare the sample, we spin coated PMMA A4 at 4000 rpm for 40 seconds and baked at 150°C for 90 seconds on hot plate. We also spin coated aquaSAVE solution which helps improve the resolution by limiting the scattering of electrons inside the resist. Aperture size was $10\ \mu m$ and EHT was set to 15 kV. The doses that worked best for 1st, 2nd and 3rd degree fractals was around 275, 285 and $345\ \mu C/cm^2$ respectively. We determined these values

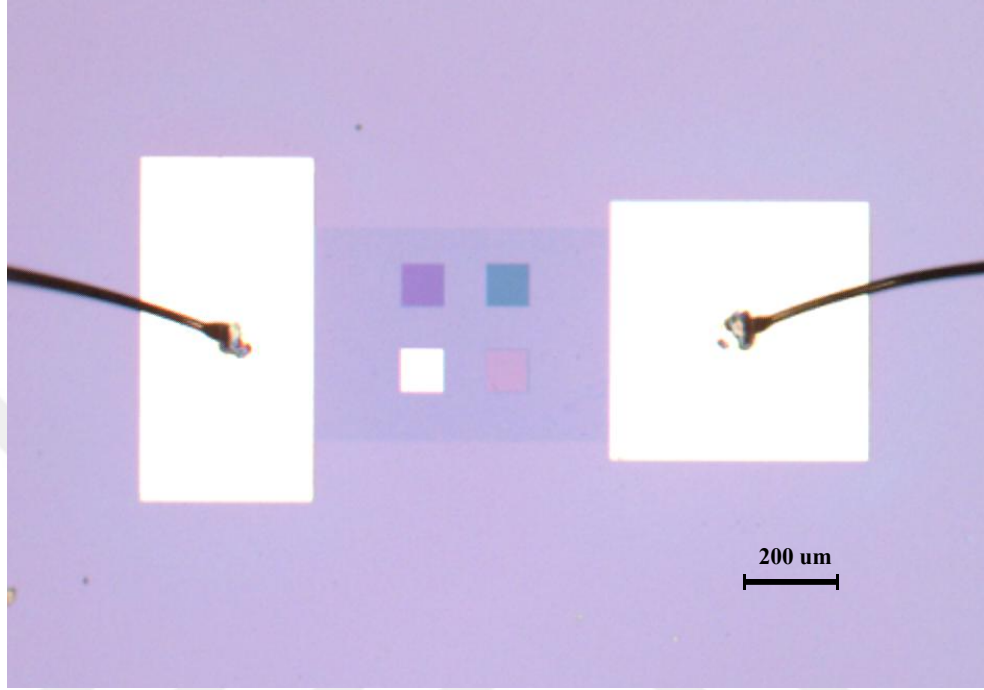


Figure 4.6: Optical microscope image of the finished device that shows graphene patch, source-drain contacts and smaller regions of fractal patterns in between.

by conducting dose tests on dummy samples and examining the features with scanning electron microscopy prior to actual device fabrication. In between each source and drain on the sample we patterned 4 active regions (see Figure 4.6). After the electron beam exposure, we developed the sample in a 1:1 MIBK:IPA solution for 40 seconds and stopped the development by putting the sample in IPA. 5/45 nm Ti/Au was deposited by electron beam evaporator and the resist was lifted-off by acetone. Figure 4.7 shows the scanning electron microscopy images of regions for each fractal degree.

4.3.5 Bonding

Next step was to separate the sample into 8 devices. This was done by spin coating a photoresist prior to dicing in order to protect the structures and dicing the sample. After that, each sample was mounted on the copper on a PCB board. The back gate was then the copper on the PCB. Two isolated copper regions on

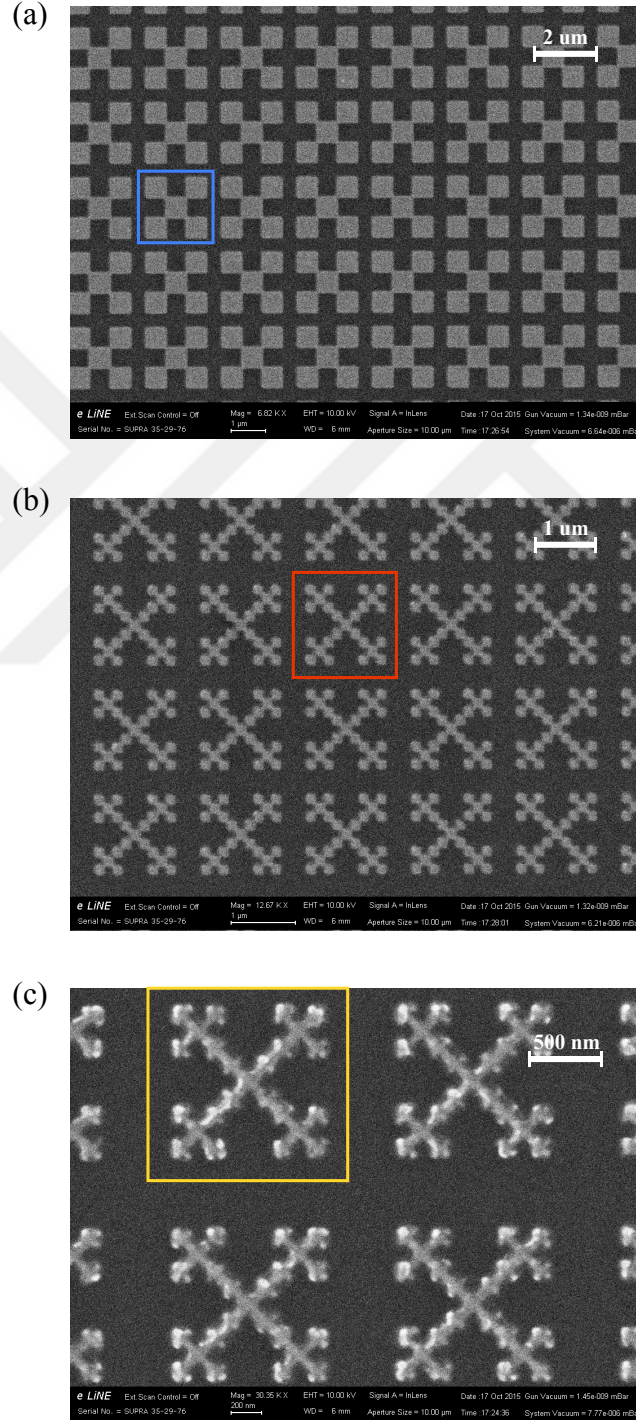


Figure 4.7: Scanning electron microscopy images of the (a) 1st, (b) 2nd and (c) 3rd degree fractal patterns. Unit cells are shown in red, blue and yellow squares for 1st, 2nd and 3rd degree fractals respectively.

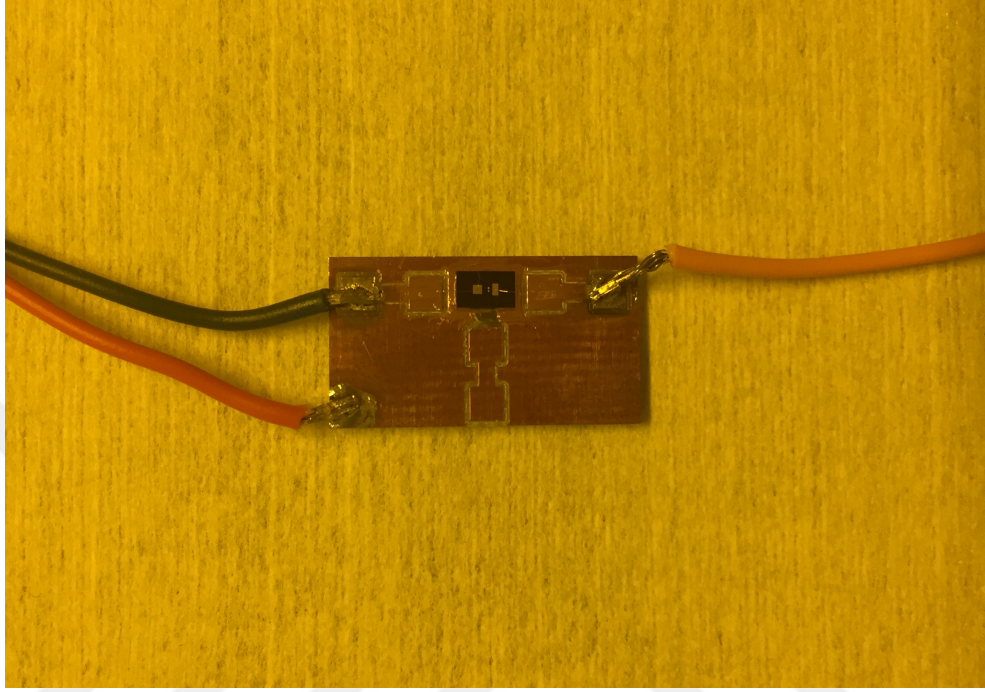


Figure 4.8: Photo of the finished back gated device.

the PCB was used to wire bond source and drain contacts. We finally soldered wires to these three contacts and the device was ready for experiments. The finished device is shown in Figure 4.8.

4.4 Fabrication of the Top Gated Device

The top gated device has two contacts; one contact taken on graphene and another from the gold of the top gate (see Figure 4.1b). The fabrication steps of this device are explained in the following subsections.

4.4.1 Fabrication of Square Fractal Patterns

Again, we started fabrication with $10\text{ mm} \times 10\text{ mm}$ CVD grown monolayer graphene on Si/SiO₂ samples purchased from Graphene Supermarket. We did

the electron beam lithography of the fractal patterns the same way as the back gated device (Refer to Section 4.3.4 for details). Only this time we arrayed $100\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$ active regions into a $2\text{ mm} \times 2\text{ mm}$ area which is equal to the size of the window of the top gate. This is to make sure that all the regions were visible when the top sample was aligned on the bottom sample. After electron beam lithography and metalization the bottom sample was ready.

4.4.2 Preparation of Top Gate

For the top sample we started with a CaF_2 substrate that is transparent in the middle infrared frequencies that we are working in. We placed a $2\text{ mm} \times 2\text{ mm}$ tape and deposited 300 nm thick Au on the sample via electron beam evaporation. After that we removed the tape and achieved a $2\text{ mm} \times 2\text{ mm}$ window opening on the sample.

4.4.3 Assembling Samples

To form the final device, we placed the top gate as gold side facing down onto the graphene sample making sure the patterned regions are aligned to the window opening. Double sided adhesive carbon tapes are used at this stage not only to leave some space between the substrate and the top gate but also as electrical contacts. Finally, we filled this space between graphene and the top gate with ionic liquid i.e., diethylmethyl(2-methoxyethyl) ammonium which is commercially available. After the assembly, the device was ready for experiments (see Figure 4.9).

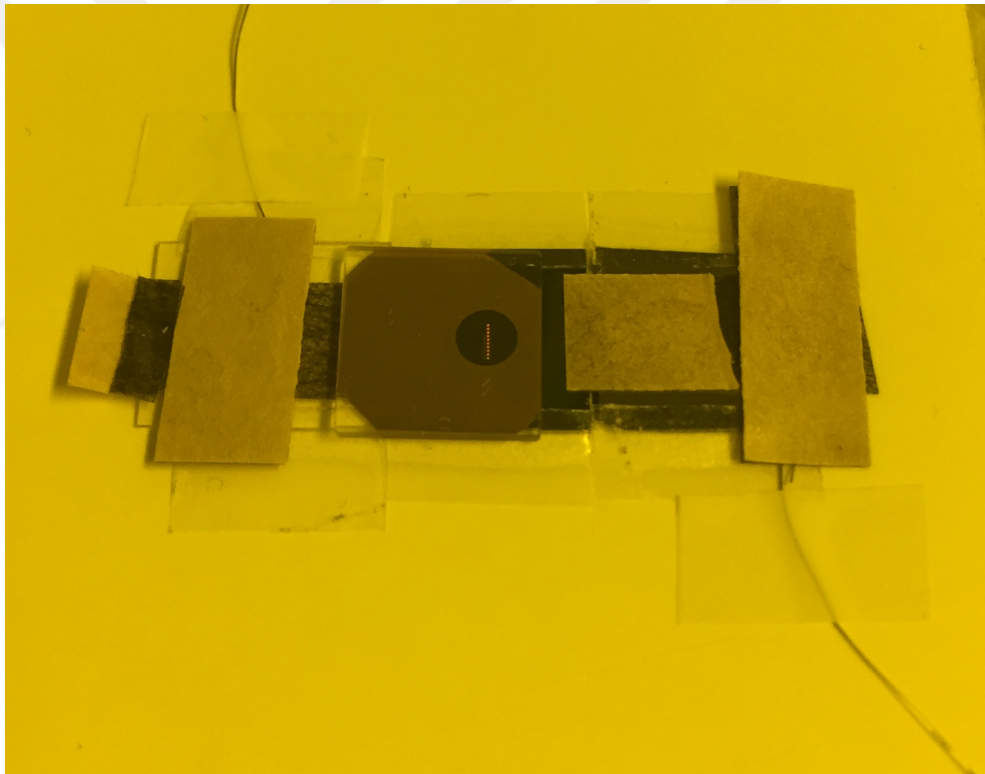


Figure 4.9: Photo of the finished top gated device.

4.5 Experiment Results

When the fabrication of both devices were complete, we conducted a series of experiments. During these experiments, reflection measurements are taken using an FTIR spectroscopy system integrated with a microscope. Using the microscope incoming light is focused on the regions of the different fractal patterns. Reflection spectra are measured for different values of the gate voltage. In the first device (see Figure 4.1a), a small voltage of 0.2 V is applied between the source and drain contacts and gate voltage is applied between the source and back contact. In the second device (see Figure 4.1b), gate voltage is applied between graphene and the top contact.

The normalised reflection spectra of the experiments of the back gated sample are given in Figure 4.10. For this device, the charge neutrality point (CNP) is calculated using the relationship given in Equation 4.1 [31], where n is the sheet carrier concentration and C is the capacitance per unit area.

$$C = \frac{ne}{V} \quad (4.1)$$

Hall measurements of the graphene samples gives the sheet carrier concentration at no applied voltage as $n = 6.4 \times 10^{12} \text{ cm}^{-2}$ therefore the capacitance per unit area is estimated as $C = 1.2 \times 10^{-4} \text{ Fm}^{-2}$ using Equation 4.1. Thus, CNP is expected to be at 85 V, and the gate voltage applied during the FTIR measurements spans a voltage range from 85 V down to -50 V. ΔV values, which are applied gate voltages with respect to V_{CNP} , are indicated in the legends of Figure 4.10 for clarity.

The normalised reflection spectra of the experiments of the top gated sample are given in Figure 4.11. The CNP for this device is found at -0.6 V with I-V measurements. Gate voltages higher than 2.5 V induces irreversible structural deformation on graphene [32]. Hence, for this sample gate voltage applied during the FTIR measurements spans a voltage range from -0.6 V to 2.5 V. Again, ΔV values, which are applied gate voltages with respect to V_{CNP} , are indicated in the legends of Figure 4.11 for clarity. In all three measurements, reflection drops

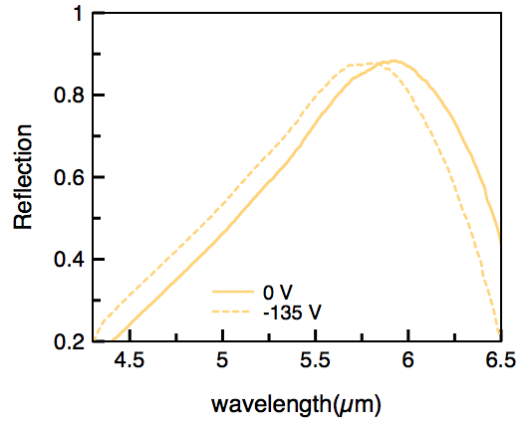
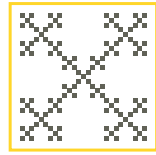
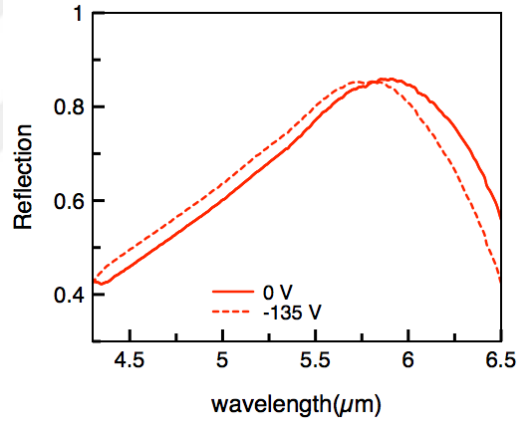
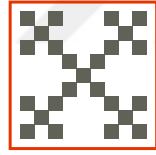
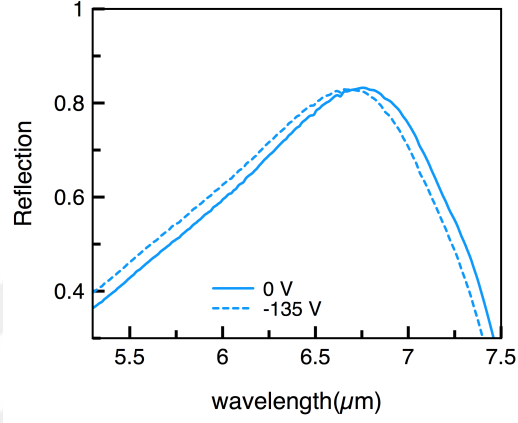
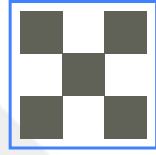


Figure 4.10: The spectral tunability for 1st (blue), 2nd (red) and 3rd (yellow) degree fractal patterns on the back gated device. In each graph, solid lines and dashed lines represent experiments with different applied voltages.

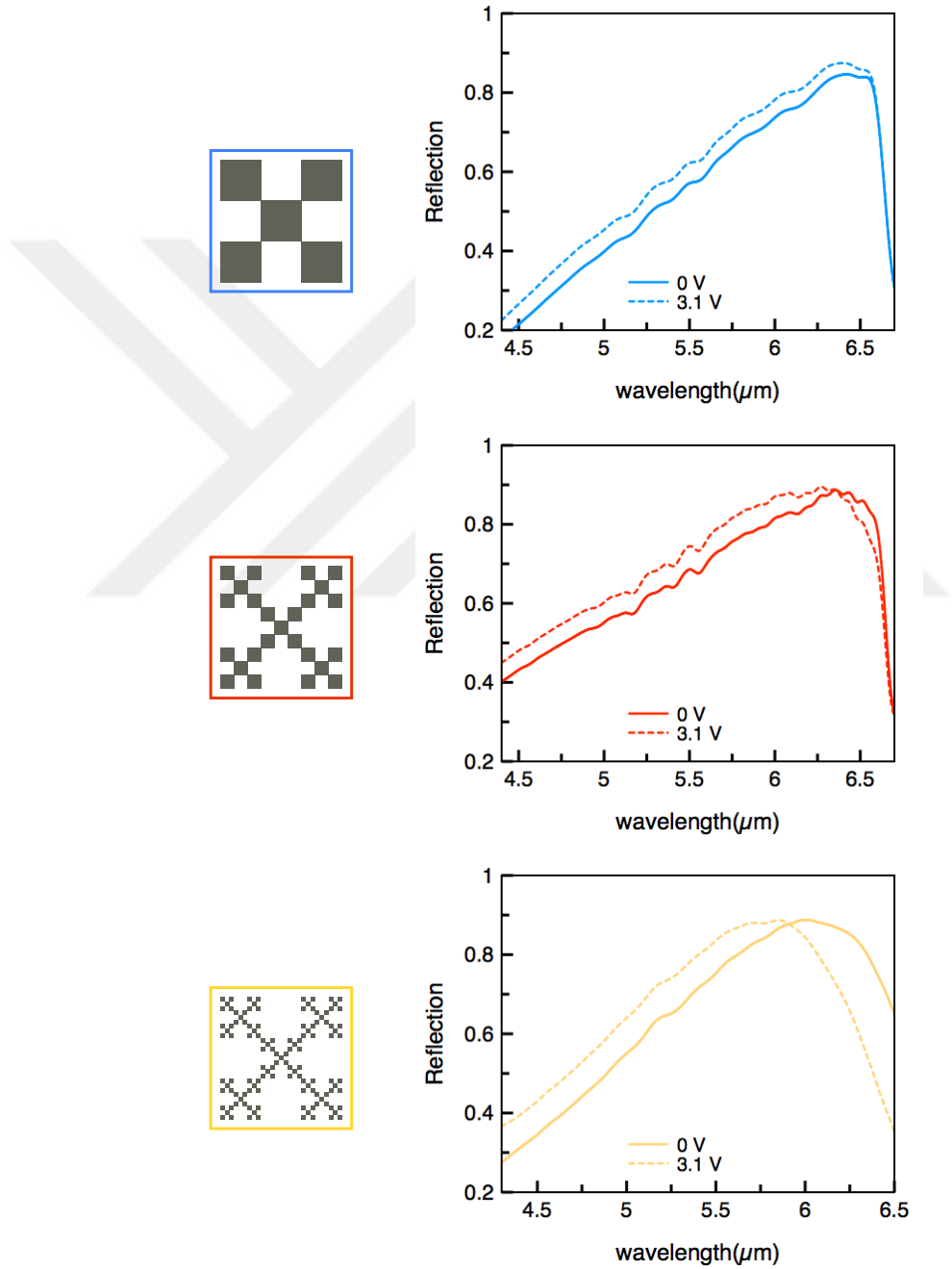


Figure 4.11: The spectral tunability for 1st (blue), 2nd (red) and 3rd (yellow) degree fractal patterns on the top gated device. In each graph, solid lines and dashed lines represent experiments with different applied voltages.

sharply at $6.5\mu m$. This is caused by the absorption of the ionic liquid electrolyte after this wavelength. It is important to design the structures such that the resonance falls inside the transparent window of this electrolyte.

For the back gated sample a voltage span of 135 V was required to achieve the amount of tunability in the simulations. For the top gated sample, however, a voltage span of 3.1 V was enough to achieve similar amounts of tunability. The top gated sample therefore is said to be a more efficient gating scheme. The simulation and measurement results also show that the amount of wavelength shift is greater for higher degree fractal structures. This relationship is due to the greater light-graphene interaction for plasmonic structures with higher degree fractal patterns as depicted in Figure 3.3. For 3rd degree fractals, electric field is more localised on the graphene due to the plasmonic modes, hence more tunability of the resonance is achieved.

Chapter 5

Conclusion

The simulation and experiment results show that electrical gating of graphene allows actively tuning the resonance wavelength. As we explained in previous chapters, plasmonic resonance wavelength strongly depends on the permittivity of the particle as well as its surrounding medium. The presence of graphene at the interface between gold squares and SiO_2 affects the plasmonic resonance in two mechanisms. Permittivity of graphene becomes an important factor because not only it affects the surrounding medium for gold surface plasmons but also graphene supports surface plasmons itself. Therefore manipulation of graphene permittivity results in a shift in resonance wavelength. Graphene permittivity strongly depends on the Fermi level and in turn the sheet carrier concentration. Hence, by gating graphene resonance wavelength can be tuned.

In addition to this, we examined the relationship between plasmonic localization and tunability of graphene. Going from 1st degree to 3rd degree fractals we showed that more electric field is confined to the volume around graphene. This due to increased number of localisation centers (corners of the squares) as well as the increase in the magnitude of the localized fields. In addition to this, we observed that spectral tunability of the reflection resonance was also greater for higher degree fractals. Therefore, we can deduce that more plasmonic field coupling with graphene results in more tunability of the plasmonic resonance.

Finally, we compared two different methods for the electrostatic gating of graphene. We fabricated two different devices with the same active regions of plasmonic structures on graphene. The first device had a transistor like layout with source and drain contacts on graphene and a back gate under the substrate. Two power supplies were required for the device operation. The second device was top gated via an ionic liquid electrolyte. Experiment results suggest that top gating is a more efficient method. FTIR measurements of reflection spectrum show that similar amounts of spectral tunability was achieved by applying a gate voltage of 135 V and 3.1V for top and back gate devices, respectively. Thus we conclude that Fermi level of graphene can be manipulated by applying much less voltages for the second device. The fabrication of the second device is also simpler compared to the first device which requires extra steps like isolation of graphene and lithography for source and drain contacts. Gating via a top contact and an ionic liquid seems like it is a more preferable way for graphene devices. It is important however to make the designs considering the transparency window of the ionic liquid for electro-optic applications. Transparency window of ionic liquid limits the frequency of operation.

To sum up, we propose an electro-optic graphene device that enables active control of reflection resonances. These devices can be used for filtering and switching applications. The shape of the resonances can be modified by designing plasmonic structures in different shapes and with different materials. Frequency of operation can also be altered by the size of the plasmonic structures however the optical properties of graphene at the desired frequency should be calculated. Tunability of graphene is different at different frequency regimes. Furthermore, transmission of light can also be modulated with similar devices as long as transparent substrates are chosen. For example, a top gated device on a CaF_2 substrate would exhibit a transmission resonance and similar tunable properties.

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