A THESIS

FOR THE DEGREE OF MASTER OF ENGINEERING

Formation of graphene flowers for field enhancement and its applications for low-voltage operation of ferroelectric polymer memory devices

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Date

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To

My Parents & Professor

Abbreviations and notations

G.F	Graphene flower
MEK	Methyl ethyl ketone
PGMEA	Propylene glycol methyl ether acetate
IPA	Iso Propyl Alcohol
MFMS	Metal-Ferroelectric-Metal-Semiconductor
MFS	Metal-Ferroelectric-Semiconductor
MFIS	Metal-Ferroelectric-Insulator-Semiconductor
MFMIS	Metal-Ferroelectric-Metal-Insulator Semiconductor

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Abstract:

As an emerging area in organic electronics, polymer memories have become an active research topic in recent years, because they are likely to be an alternative or supplementary technology to the conventional memory technology facing the problems and challenges in miniaturizing from microscale to nanoscale. Here we propose the device architectures based on ferroelectric polymers particularly emphasizing on the device elements such as metal/ferroelectric/graphene/metal type capacitor with ferroelectric poly(vinylidene fluoride) (PVDF) and its copolymers with trifluoro ethylene (TrFE).

Different solution processing methods were used for the growth of graphene flowers film over silicon and gold coated silicon. Using these different fabrication techniques, we observed density, uniform dispersion of graphene nano particles. Moreover, detailed morphological studies were carried out by SEM to check the spreading density and uniformity of graphene flowers on various substrates. The physical properties of graphene flowers coated on various substrates were investigated by Raman spectroscopy. A comparative study was performed with already reported results by Lin Li et al., to demonstrate efficiency of our proposed device architecture to minimize operating voltage of polymer memory. So, by comparing our obtained sample morphology with their graphene floral structure, it almost most looks similar with even more sharp edges than their sample. Hence, can say that our grown graphene flowers are efficient enough for the high field emission required for polarization reversal of polymer memory device at low voltage operation

1. Introduction

1.1 Research Background

Information technology (IT) has played an important role in our lives and now it has become an its essential part. Information Technology in the form of many portable and non-portable electronic devices, e.g. mobile phones, personal computers, person digital assistant (PDA), fax machines, media players and many more, can be seen anytime and everywhere. As the complexity of these mobile gadgets increases, miniaturization and data storage become key issues. All IT gadgets require data storages or memories. Conventional memories are implemented on semiconductor-based integrated circuits, such as transistors and capacitors. Since the demand for mobile applications is the main driving force behind memory technologies and devices, there is an ever-increasing demand for higher capacity and system performance, lower power consumption, smaller form factor and lower system cost.

In electronics, a memory usually refers to a component, device or recording medium that retains retrievable digital data over a time interval. It is one of the fundamental components of all modern computers and electronic systems [1]. An electronic memory is fast in response and compact in size and can be read and written when connected to a central processing unit.

Electronic memories can be divided into two primary categories according to its volatility: volatile and nonvolatile memories. Volatile memory loses the stored data as soon as the system is turned off. It requires a constant power supply to retain the stored information. Non-volatile memory can retain the stored information even

when the electrical power supply has been turned off. They can be further divided into sub-categories, as shown in Fig. 1, with the write-once read-many times (WORM) memory, the hybrid non-volatile and rewritable (flash) memory, and the dynamic random-access memory (DRAM) being the most widely reported polymer memories.

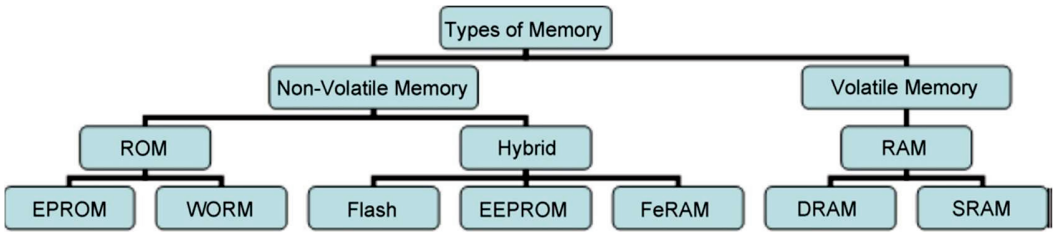


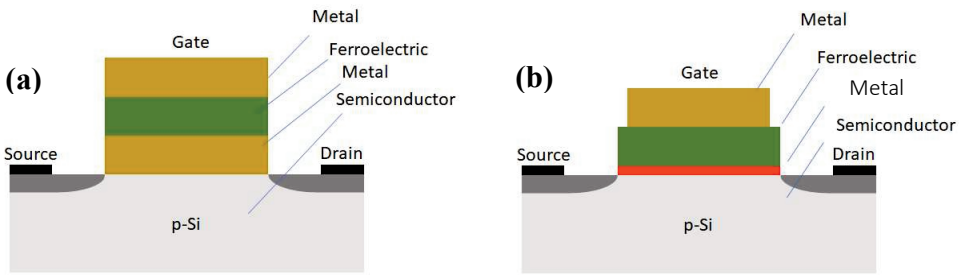
Fig. 1. Classification of electronic memories [3]

There are several current technologies providing some form of nonvolatile random-access-memory (NV-RAM) data storage to meet these needs [2]. Currently, the most widely used nonvolatile memory is Flash, a charge-storage technology that can achieve relatively high density at low cost because it is based on standard silicon integrated-circuit technology. The main drawbacks of Flash memories are low speed, cycle energy usage, and limited cycle life. Other candidate non-volatile random-access memory (NV-RAM) technologies currently under consideration include magnetic random-access memory (MRAM), Ovonic Unified Memory (OUM), Resistive random-access memory (RRAM), and ferroelectric (FRAM) [3]. The new memory technologies are based on electrical bi-stability of materials resulting from changes in certain intrinsic properties such as magnetism, phase, conformation, conductivity and polarity in response to the applied electric field.

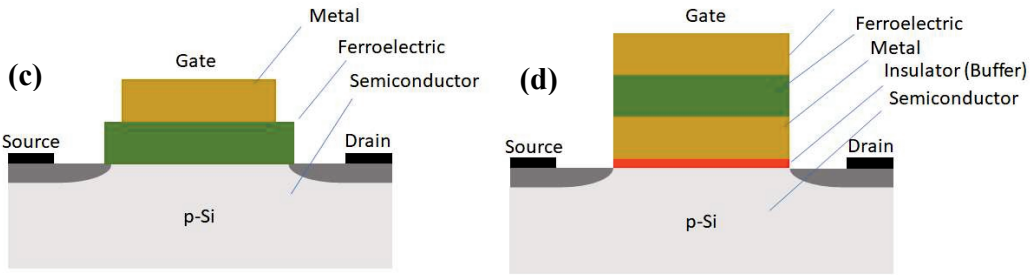
In 1970, polymer electronic memories were first reported by Sliva et al. [4], from devices based on Saran® wrap, a thermoplastic resin derived from poly(vinylidene chloride) and polystyrene. Reproducible, bi-stable switching was also observed by Segui et al. in polymer films prepared by glow-discharge polymerization. Following these pioneering works, a wide variety of polymers prepared by glow-discharge polymerization [5–7], electron-beam deposition [8], or plasma polymerization [9] have been reported to show threshold and memory switching effects.

In terms of device architectures, the polymer memories can be also classified by 3 main categories such as capacitor, transistor and resistor [10]. Recent emerging interest in ferroelectric polymers for non-volatile memory application is obvious. Various design structures for FeFETs gate stack layouts and various buffer layer configurations have been developed as shown in Fig.2:

- MFMS: Metal-ferroelectric-metal-semiconductor
- MFS: metal-ferroelectric-semiconductor
- MFIS: metal-ferroelectric-insulator-semiconductor
- MFMIS: metal-ferroelectric-metal-insulator semiconductor



MFMS MFIS



MFS MFMIS

Fig.2. Gate stack layouts and various buffer layer configurations

1.2 Research Purpose

The recent studies have extensively focused on understanding the fundamental memory properties of metal/ferroelectric thin polymer film/metal (MFM) capacitors for the potential application of non-volatile 1T-1C memory cells. The issues involved are operating voltage, remnant polarization, fatigue, thermal budget, imprint of a hysteresis curve, switching speed and data retention. The low operating voltage is one of the most important factors and thus makes our attention to the capacitors with the ferroelectric PVDF and its copolymers with TrFE films at least less than 200 nm in thickness due to their relatively high coercive field (*Ec*) of approximately 50 MV/m. A capacitor is operated at the lower voltage when the ferroelectric film becomes thinner and thinner. A MFM capacitor prepared with cyclohexanone solvent exhibited a fully saturated polarization hysteresis curve with the sweep voltage of ±15 V due to the near defect-free film arising from the solvent [11]. The low voltage operating MFM capacitor with a P(VDF-TrFE) film at ±12 V was achieved by employing thin polypyrrole-poly(styrene sulfonate) acid film as interface layers between both bottom and top metal electrodes [12]. A recent work by Naber et al has also demonstrated a MFM capacitor with a P(VDF-TrFE) layer operating at 10 V

when a PEDOT;PSS layer is inserted between bottom electrode and the ferroelectric polymer [13]. Despite the concern of the rapid reduction of the degree of crystallinity at a film thinner than approximately 100 nm [14], the recent work by Ishiwara et al. [15] has demonstrated a MFM capacitor with a 60 nm thick P(VDF-TrFE) film of approximately

1.2.1 **Proposed Device Architecture**:

We propose a device architecture of polymer memory device in which graphene flower (nano structures of graphene with sharp edges) layer is sandwiched between Metal and Ferroelectric layer. This graphene layer can generate strong electric field. This electric field is generated even in low voltage. Strong electric field can be used for ferroelectric inversion in ferroelectric capacitive memory device. Our focus is the low voltage operation of polymer memory device with graphene flowers layer sandwiched b/w metal and ferroelectric layer.

The challenge in the research was the growth of uniform graphene flowers nanoparticles film with highly dense dispersion on SiO_2 /Au substrate with thickness of less than 100 nm. We used different techniques for the growth of graphene flowers over substrate.

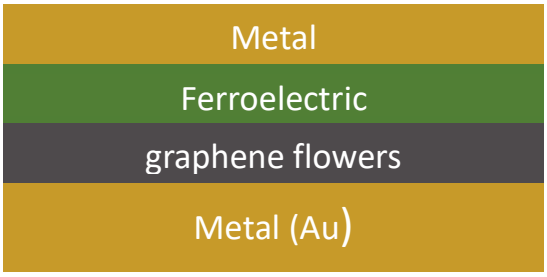


Fig. 3 Proposed Device Architecture

1.3 Ferroelectric Switching

Ferroelectricity is a spontaneous electric polarization of a material that can be reversed by the application of an external electric field A ferroelectric material exhibits a polarization–electric field-hysteresis loop (Fig. 5), analogous to the magnetization–magnetic-field-hysteresis loop exhibited by a ferromagnetic material. In addition, like the magnetic material, ferroelectric material can form domains, each with a unique polarization axis [16]. As shown in Fig. 5, when voltages are applied from 0V to $+V$cc and from 0V to $-V$cc, the polarization state changes progressively around the loop from point A to B to C, and point D to E to F, respectively. When voltages are applied from $+V$cc to 0V and from $-V$cc to 0V, the polarization state changes, moving from point C to D and from point F to A, respectively.

Under this situation, the amount of polarization charges decreases slightly, without reversing its direction [17]. Thus, the hysteresis loop is characterized by the magnitude of the zero-voltage remnant polarization (P_r, points A and D) after saturation with a large voltage (V_{cc}) and by the magnitude of the coercive voltage (V_c), the minimum value of the voltage necessary to reverse, or switch, the polarization state. To apply these ferroelectric characteristics to a memory, the two net stable states, “upward polarization” and “downward polarization”, are defined as “1” and “0” signals [18]. The coercive field V_c, is closely related to the operating voltage of the memory. The maximum voltage width of hysteresis curve is known as memory window.

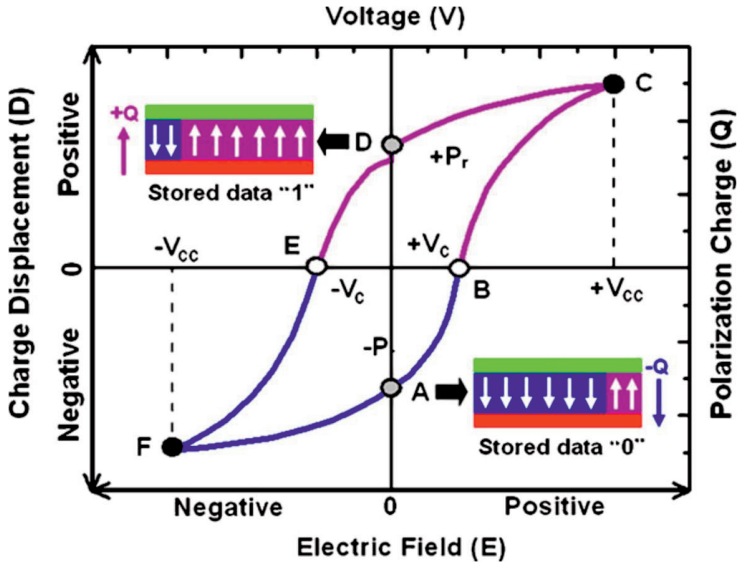


Fig. 4 Charge displacement–electric field (*D–E*) hysteresis loop and ferroelectric capacitor polarization conditions [3].

2. Experiment

2.1 Sample Preparation

We used many solution processing methods for the growth of graphene flowers on substrate. These different methods include:

- Spin coating of graphene flower solutions in two solvents methyl ethyl ketone (MEK) and Propylene glycol methyl ether acetate (PGMEA) over SiO_2 wafers
- Sonication of graphene flower and its coating on SiO_2
- Spray coating graphene flower Solution over substrate covered with Shadow Mask
- Sliding method, i.e. coating G.F(MEK) over two wafers and then sliding each one over each other
- Spray coating diluted G.F(MEK) many times by placing substrate on hot plate

- Preparation of surfactants & graphene flower solution and spin coating over SiO_2 substrate

2.1.1 Spin coating of graphene flower

We have graphene flowers solution in two different solvents, one is methyl ethyl ketone (MEK) and the other one is propylene glycol methyl ether acetate (PGMEA). We used different RPMs to spin coat graphene flower solution over the substrate. Different rpms resulted in different density of graphene nanoparticles on the substrate. It was observed that as a result of low RPM spin coating, graphene flower particles were collected together to form a bump like shape. We also tried high RPM like 2000 and 2500 but at these higher RPM, the density of graphene nanoparticles was less than the density obtained at low RPM i.e., 500 and 1000.

So, we optimized RPM and rotation time of spin coating as 1500 and 10 seconds respectively. Soon after spin coating samples were heated at 80 °C to evaporate the solvent from the substrate, as the boiling point of MEK is 79.64 °C. We also tried the spin coating of graphene flower solution in PGMEA solvent over substrate using different RPM. Then we hated our samples add 150 °C because of high boiling point of this solvent as compared to MEK. This combination graphene flowers and PGMEA solvent was not effective as compared with previously used combination. Schematic illustration of spin coating process is demonstrated in figure below.

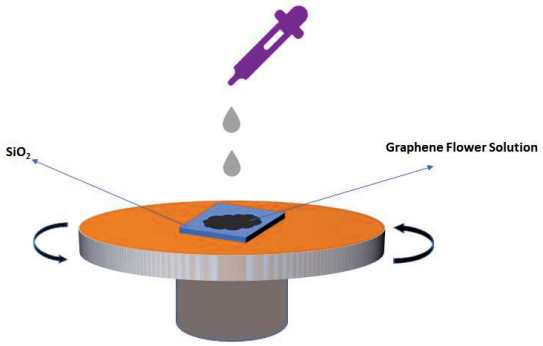


Fig. 5. Schematic of Spin coating of graphene flower

2.1.2 Sonication of graphene flower and its coating on SiO_2

In this experiment we used sonicator for the ultrasonication of graphene flower solution. At first substrates were cleaned using distilled water, IPA and acetone. then these samples were dried in oven. then we put these dry samples in the substrate carrier and Then we placed graphene flower solution in sonicator for ultrasonication for about 30 minutes. After ultrasonication of graphene flower solution we spin coated it on the substrates and then heated them.

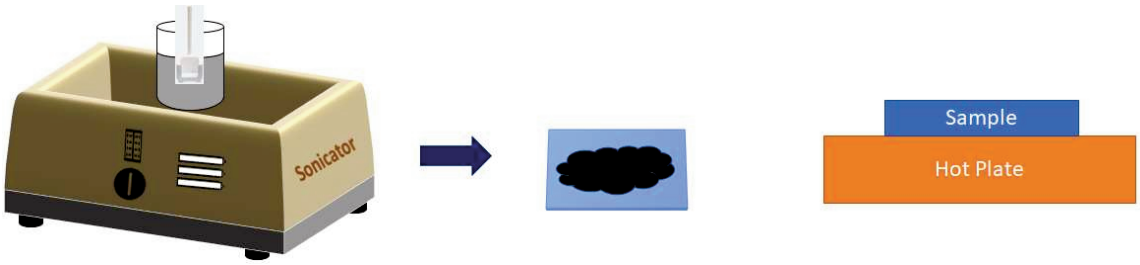


Fig. 6. Schematic of Sonication of graphene flower solution, coating it on substrate and then heating at hot plate

2.1.3 Spray coating graphene flower solution over substrate covered with shadow mask

In this experiment we used shadow mask as shown in figure .7 , the pore size of this mask was 200µm. WE used custom made spraying system to spray graphene flower solution on the substrate through this shadow mask. Spray system has a pump to push solution to spray gun and then by pushing pump the graphene flower solution ejaculates from the nozzle of spray gun in the form of small particles. When this spray plume falls on the shadow mask it goes through its pores and gets deposited over the substrate surface.

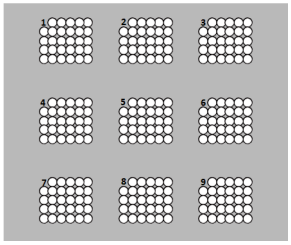


Fig. 7 Schematic of Shadow mask

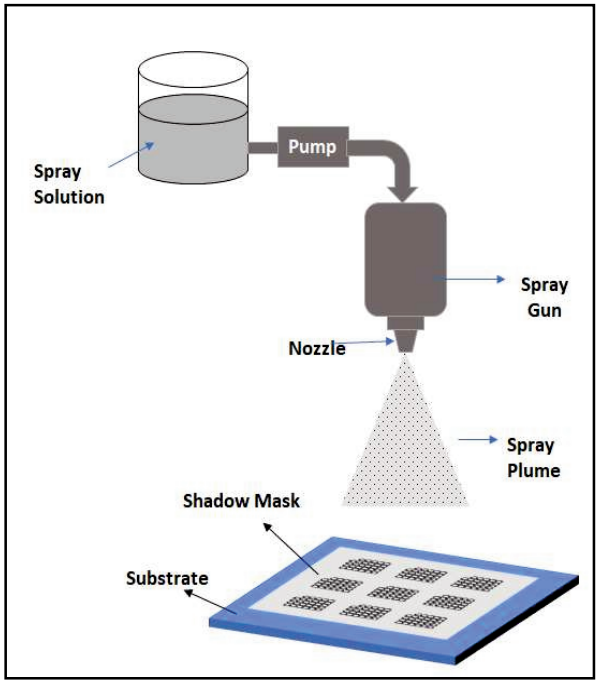


Fig. 8 Spray coating graphene flower solution over substrate

2.1.4 Sliding method

In this method graphene flower solution in MEK solvent was prayed over the two substrates. These substrates were first cleaned using distilled water, IPA and Acetone. The purpose of cleaning was to remove any kind of dust particles, finger prints and other undesired patterns from the surface of substrates. After spraying it on substrates, both were rubbed against each other with surfaces face to face as shown in fig. 9

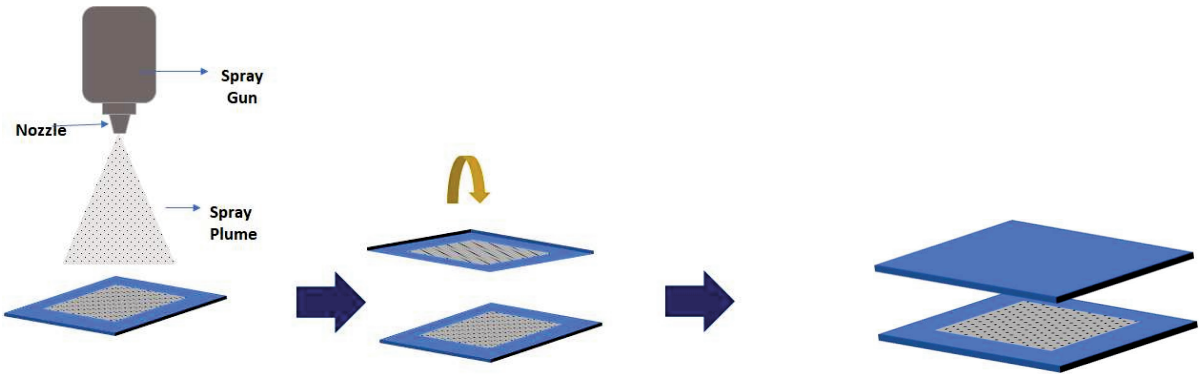


Fig. 9 Schematic of Sliding method, i.e. coating G.F(MEK) over two wafers and then sliding each one over each other

2.1.5 Spray coating diluted G.F(MEK) many times by placing substrate on hot plate

In this experiment we used same custom-made system as described above. By placing our substrate over hot plate with temperature equal to 80°C, graphene flower solution was sprayed over the substrate. This value of temperature was selected to evaporate the solvent from substrate surface instantly.

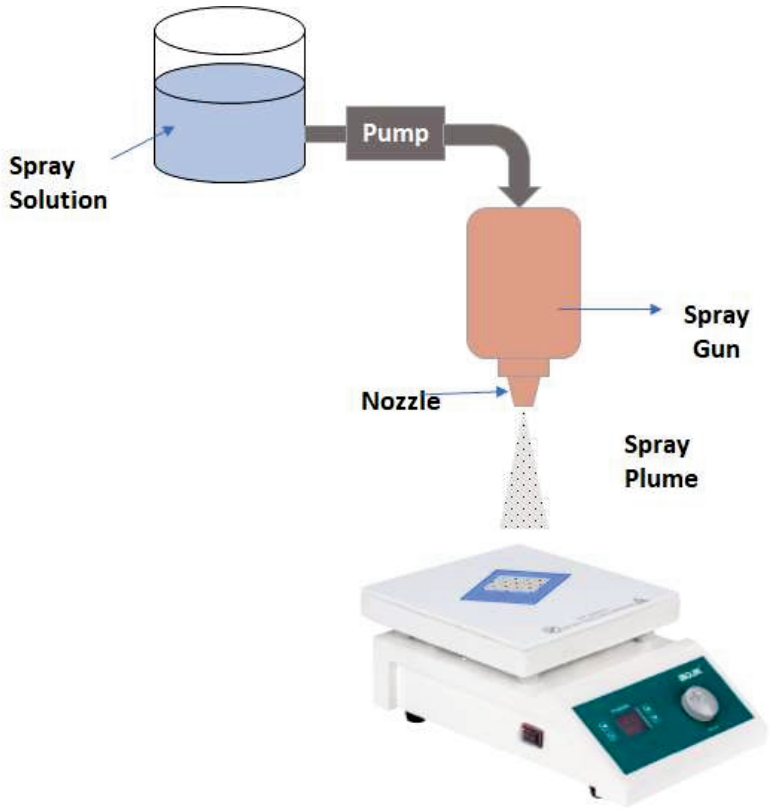


Fig. 10 Schematic of spray coating graphene flower solution over substrate covered with Shadow Mask

2.1.6 Spin coating surfactants & graphene flower solution over SiO_2 substrate

In this method, we used four combination of different solvents by mixing them in equal proportion. These different solvents include:

- Methyl Ethyl Ketone (MEK)
- Propylene glycol methyl ether acetate (PGMEA)
- Isopropanol (IPA)
- Ethylene glycol
- Ethanol amine

We mixed graphene flowers solution with these different solvents and placed on hot plate at 40 °C with continuous stirring for about 30 minutes. After this mixing process we obtained homogeneous graphene flower solution. Then these solutions were spin coated over substrate and the results were observed using optical microscope.

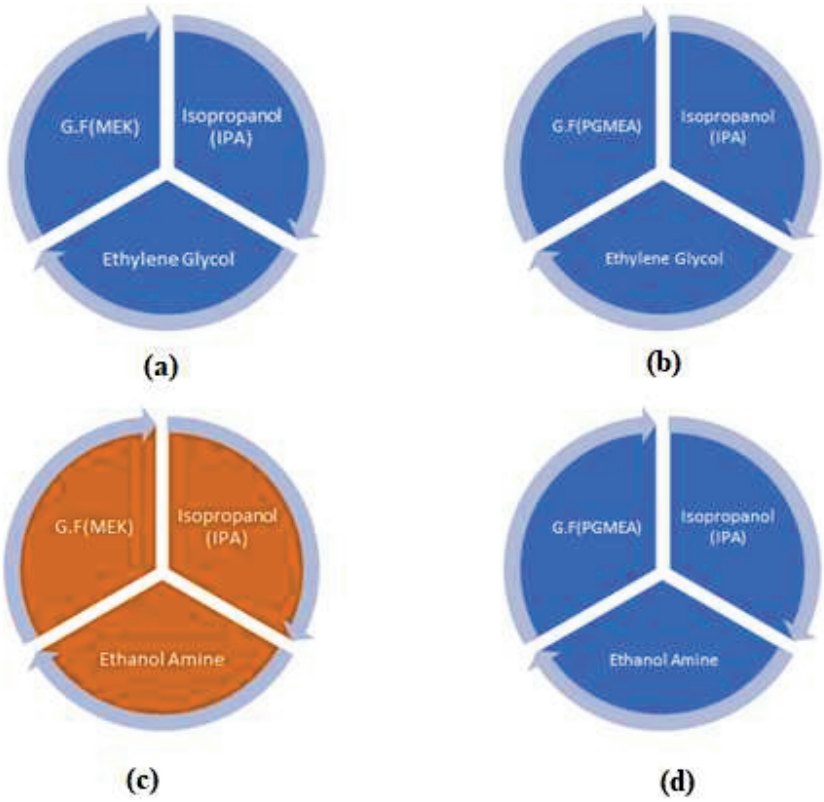


Fig. 11 Surfactants & graphene flower solution in four different combinations

2.1.7 Placing the sample on the lean holder (coffee stain phenomenon)

2.1.7.1 Coffee stain phenomenon:

When a droplet dries on a surface, the particles suspended in it usually deposit in a ring-like pattern, leaving a stain or residue, called the coffee-ring effect. Or when a solution or a particle containing suspension droplet dries, the solute is often distributed on the substrate in interesting ring patterns, usually in the vicinity of a (pinned) contact line. The rate of evaporation is higher near the periphery of the

drop which also causes a flow of liquid and particles or solute towards the contact line or periphery. The solute concentration rises near the periphery and deposit there first. Most of the polymer deposits near the periphery of the drop.

Fig. 12 Schematic of Coffee-ring effect[8]

Method:

In this method substrate was placed over the lean holder at an angle of 30 degrees. This sample over the lean holder was placed on the hot plate at temperature of 80 °C. In this way the lower part of sample was able to get more heat and it was hotter as compared to other end. If the sample is placed on the lean holder Fig.13 (a) the graphene flower drop will be formed as Fig. 13(b). The point A will be hotter than point B, so G.F flake will be precipitated from point A. As the solvent evaporates, the whole size of drop will decrease Fig. 13 (c) Then, the graphene flower bump is formed beside the previous G.F flakes. So, final formation will be shown as Fig.13(d).

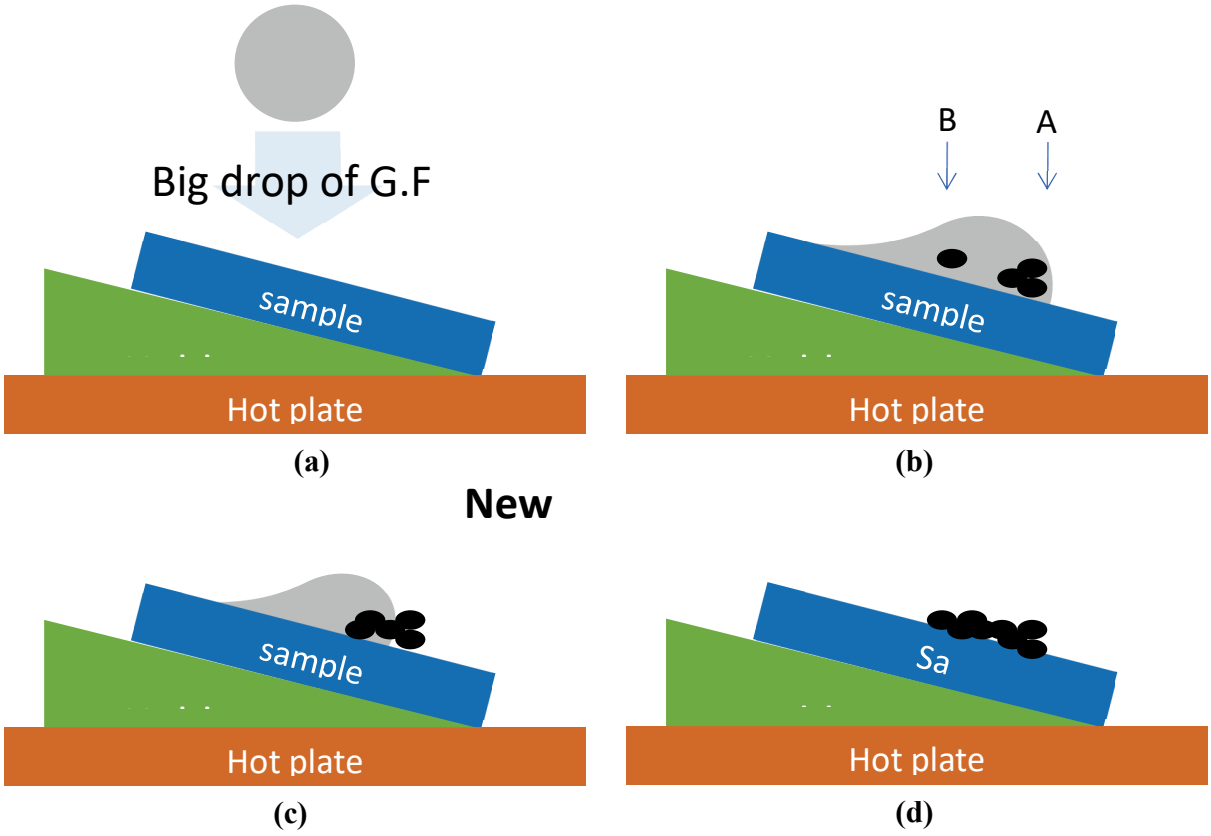
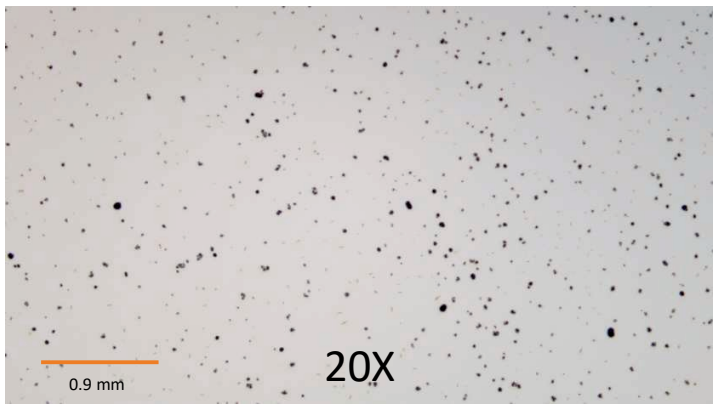


Fig. 13. Schematic of Placing the sample on the lean holder (coffee stain phenomenon)

3. Results and Discussion:

3.1 Sample Fabrication Result:

3.1.1 Spin coating of graphene flower:



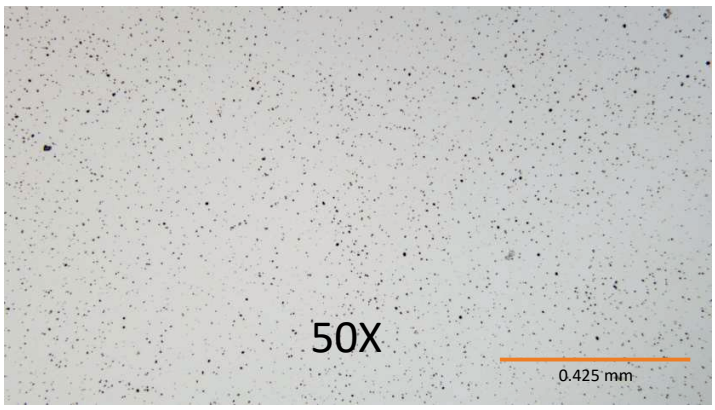


Fig. 14 (a, b). Optical microscope image of samples after Spin coating of graphene flower at different magnifications

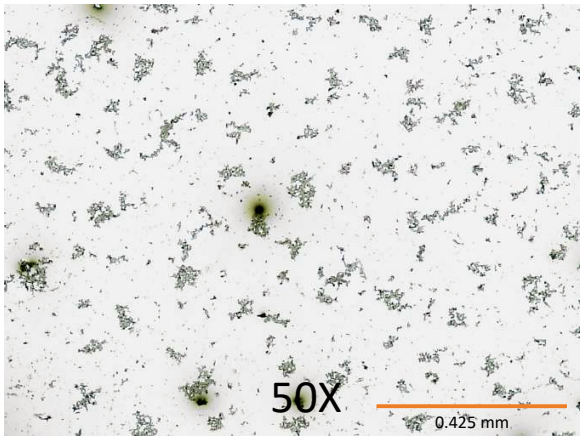
3.1.1.1 Analysis

Spin coating of graphene flower solutions in both solvents MEK and PGMEA was performed over SiO_2 wafers. After spin coating at 1500 RPM for 10s, the samples were heated. It was observed that the MEK solvent evaporated at room temperature from sample surface, while the PGMEA Solvent evaporated from sample surface upon heating because of its high boiling point then MEK solvent.

3.1.1.2 Summary

We found the graphene flowers particles over substrate, but the density was low to be used in our device structure.

3.1.2 Ultra-sonication of graphene flower solution



(b)

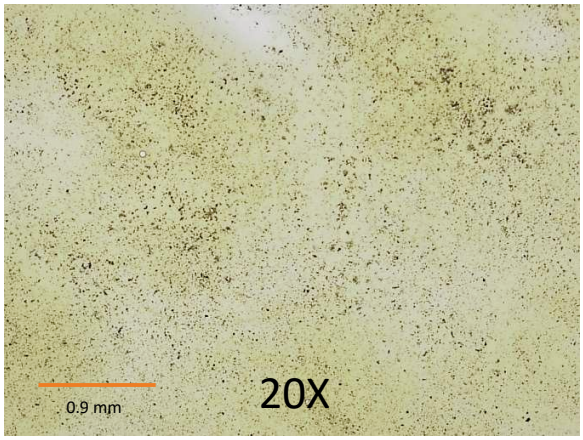


Fig. 15 Optical microscope image of samples after ultra-sonification

3.1.2.1 Analysis

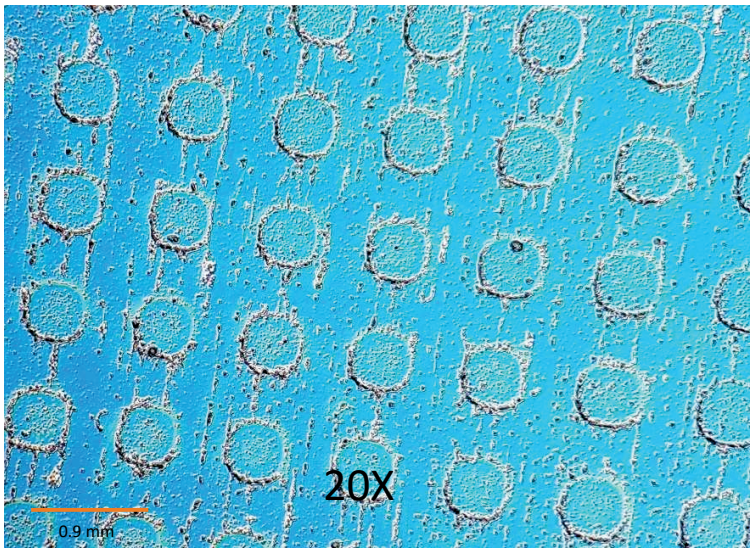
We performed sonication of our graphene flower solution, which is the act of applying sound energy to agitate particles in a solution We took coatings of the solutions over substrates. and samples were observed using optical microscope. The graphene nanostructured particles instead of getting collected to form bump like structure.

3.1.2.2 Summary

It was found that graphene flowers particles showed good dispersion over substrate, and obtained low density but still higher than from previously spin coated sample

3.1.3 Spray coating graphene flower solution over substrate covered with shadow mask

(a)



(b)

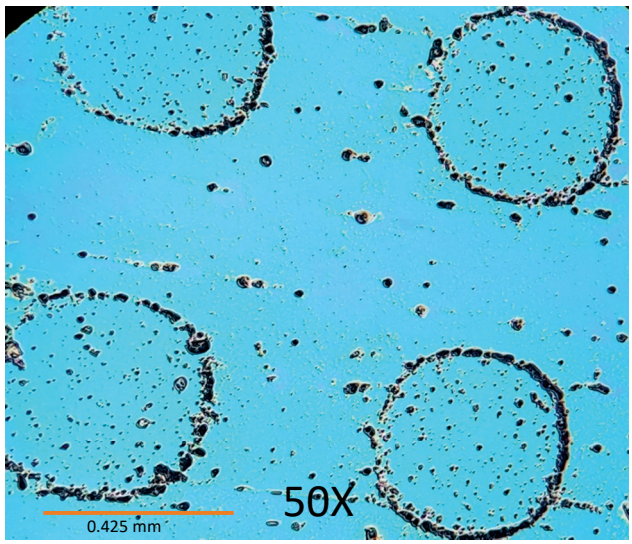


Fig.16. Optical microscope image of samples after Spray coating of graphene flower

3.1.3.1 Analysis

In our experiment, we used shadow mask with very thin holes as shown in fig. 8 The idea was to obtain a very small size graphene flower nanoparticles clusters in the specific order closely located to each other. After spraying solution over shadow mask, it was observed that graphene flower solution formed “coffee-ring” like patterns. In which nanoparticles were accumulated on the boundaries of very small holes of shadow mask

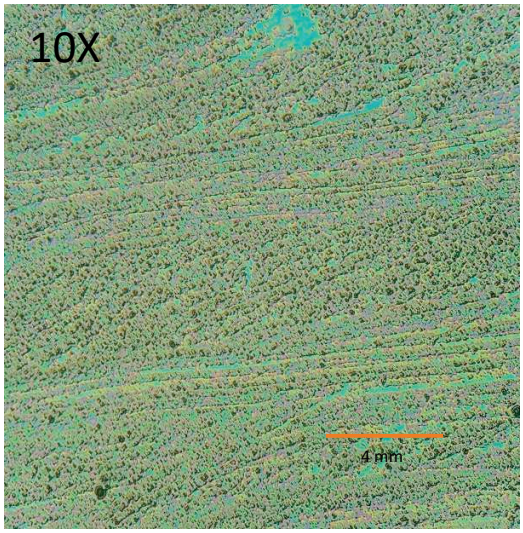
3.1.3.2 Summary

This method didn’t show up with our expectations

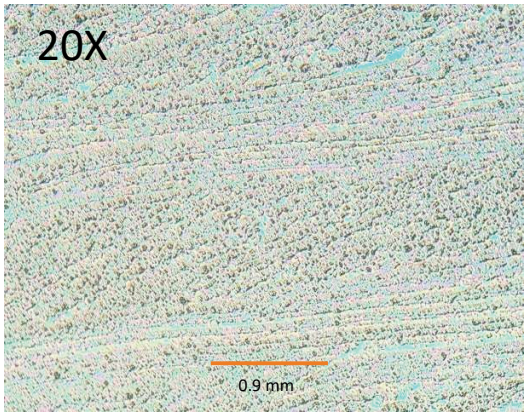
3.1.4 Sliding Method

3.1.5

(a)



(b)



(c)

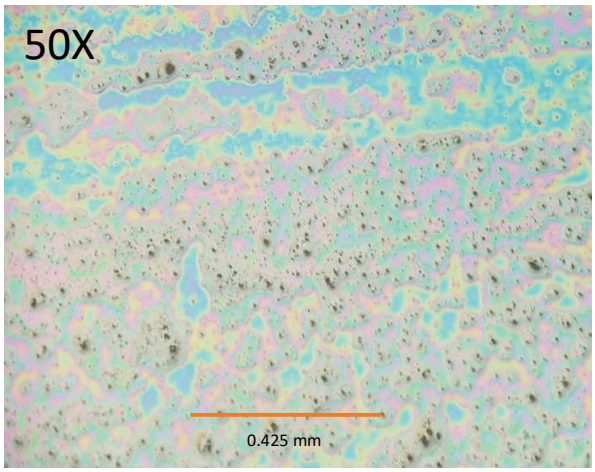
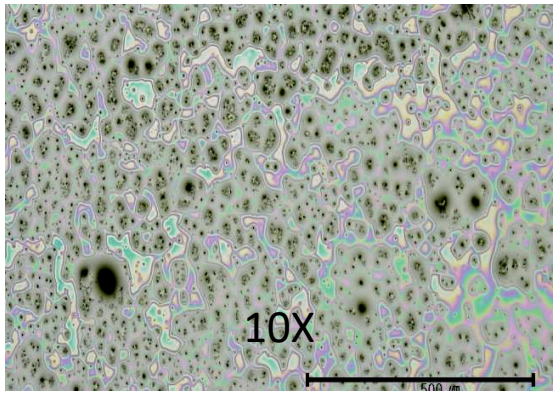


Fig.17. Optical microscope image of samples after sliding of graphene flower coated substrates at different magnifications

3.1.6 Spin coating surfactants & graphene flower solution over SiO_2 substrate

(a)



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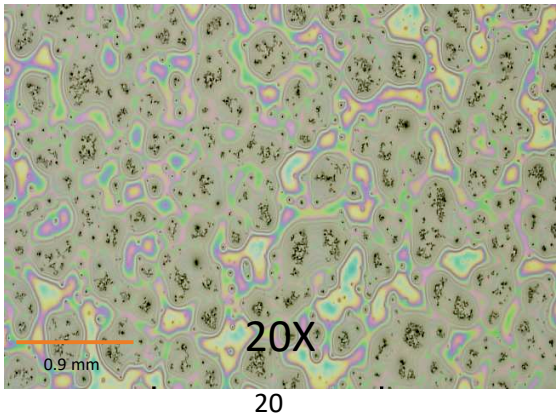


(c)

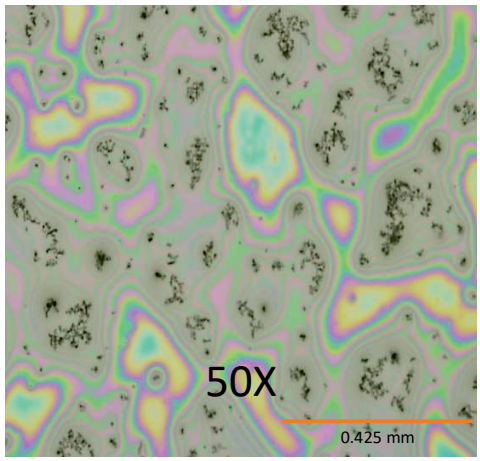


Fig.18. Optical microscope image of samples after solvent treatment of graphene flower solution and its coating over substrates at different magnifications

3.1.6.1 Analysis

We mixed different surfactants with graphene flower solution because of their inherent property to surround nanoparticles.

It was expected that because of the adhesiveness of these surfactants with substrate will result in better dispersion of nanoparticles.

3.1.6.2 Summary

Solution with combination (G.F(MEK) + IPA + Monoethanolamine)) resulted in uniform dispersity over SiO_2 wafer as compared with previous experiments Fig. (18)

3.1.7 Spray coating diluted G.F(MEK)

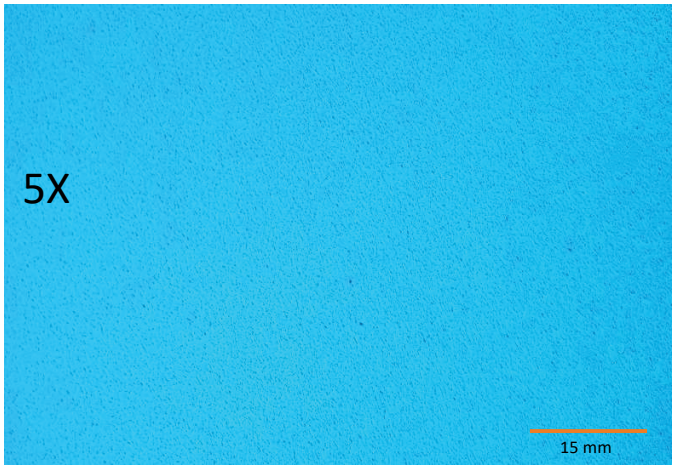
3.1.7.1 Analysis

Using custom made spraying system, graphene flower solution in MEK solvent was sprayed over substrate and it was heated on hot plate, because of low boiling point of MEK solvent, it evaporated and only graphene nano particles were deposited over substrate. A small diameter nozzle allowed small particles of graphene flowers to be easily ejected from it

3.1.7.2 Summary

Using this method, we found high density, uniform dispersion of graphene nano particles as compared with our all previously tried methods

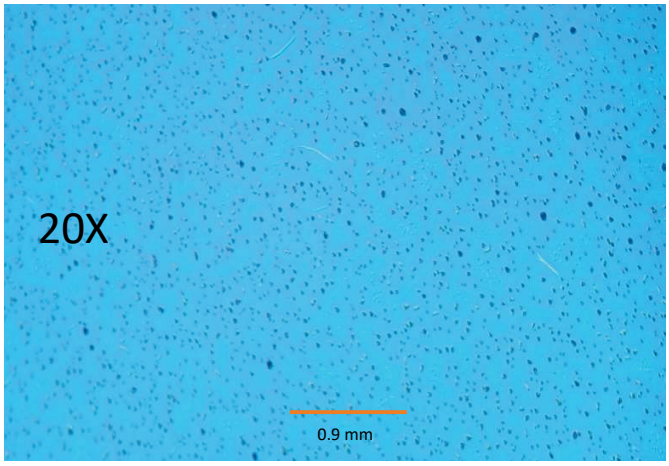
(a)



0

(b)

(c)



(d)

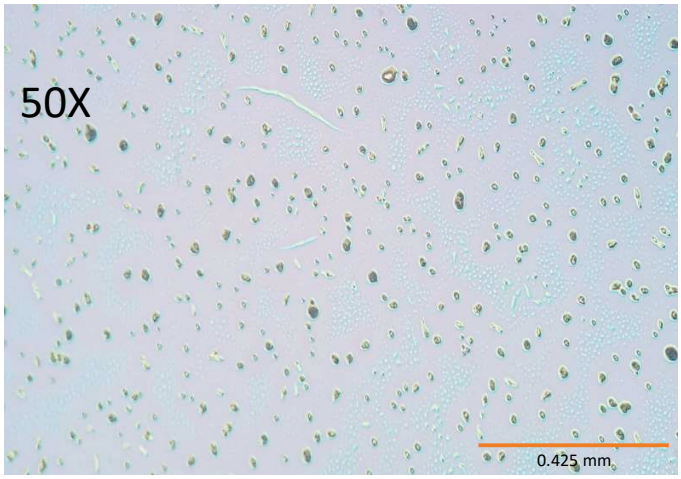
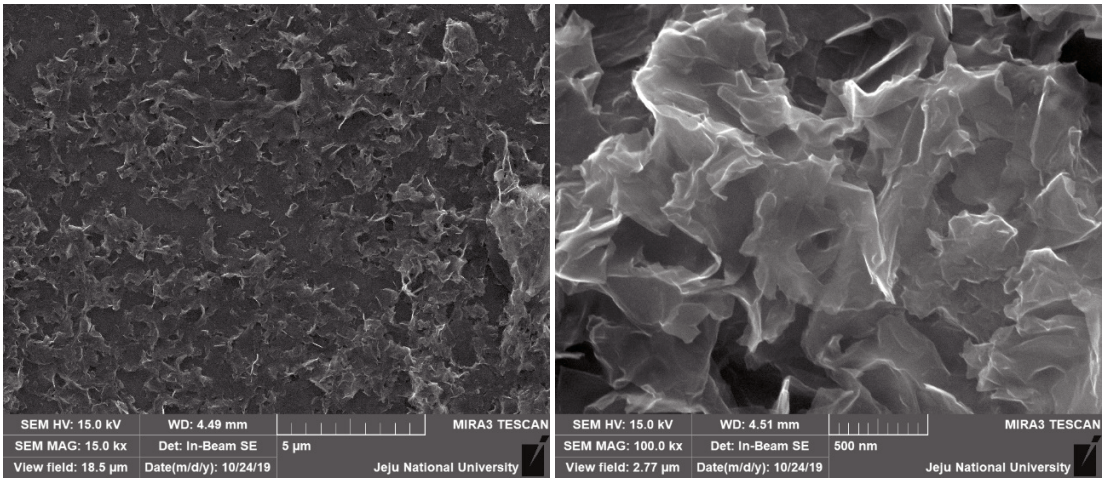


Fig. 19 Optical microscope image of samples after spray coating graphene flower over substrates at different magnifications

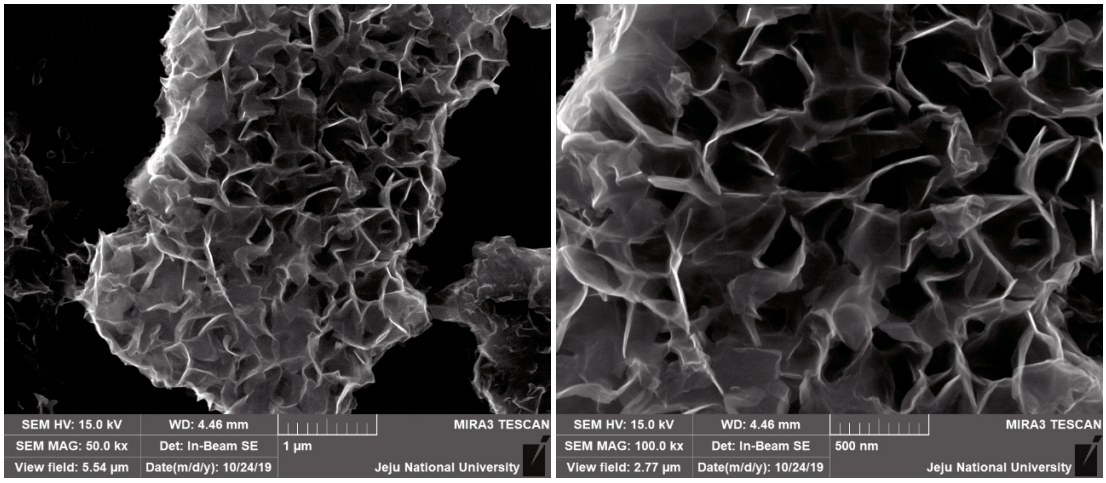
4. Characterizations

4.1 Sample Morphology and Chemical Structure

Fig. 21 shows SEM image of graphene flowers coated over SiO_2 substrate.



(a) **(b)**



(c) **(d)**

Fig. 20. SEM Images of graphene flower coated over SiO_2 sbstrate

As shown in figure, the bright regions are the edge of graphene flowers, thus flower-like formation can be seen.

Significant enhancement of graphene's electron field emission can be obtained only in the sharp edges of graphene when an electric field is applied along the sheet because of the reduction of the work function at the edges as compared to the graphene sheet body [19].

4.2 A Comparative Study

Lin Li et al., studied field emission properties of graphene sheet with open surface and sharp edges structure [20]. Fig. shows the SEM image of their grown flower like structure. They also reported enhanced electron field emission due to the high conductivity and abundant sharp edges of Floral-clustered graphene structure (Fig.22). The obtained turn-on electric field (E_{on}, 2.6 V μm^{-1} at 10 μA cm^{-2}) was also significantly lower

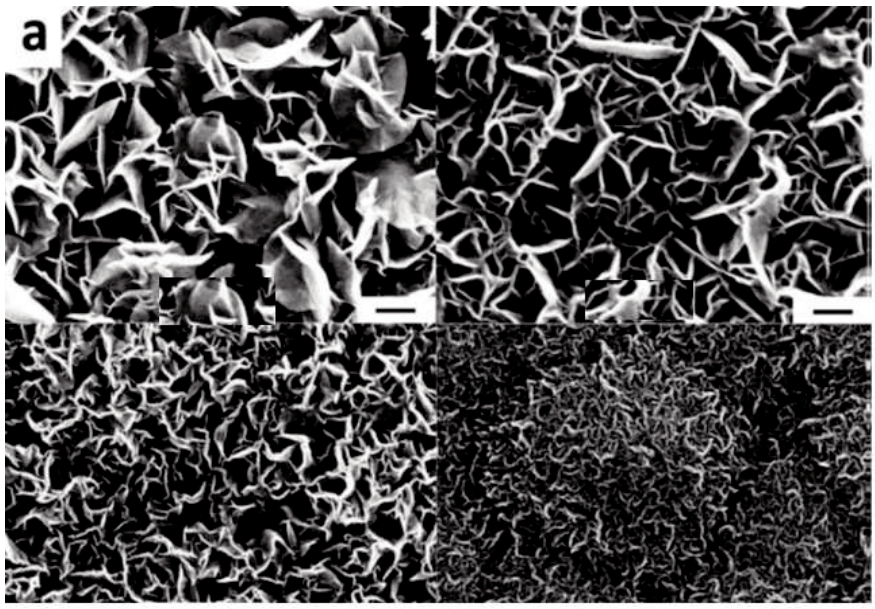


Fig.21 SEM image of Floral-clustered few-layer graphene nanosheet [22]

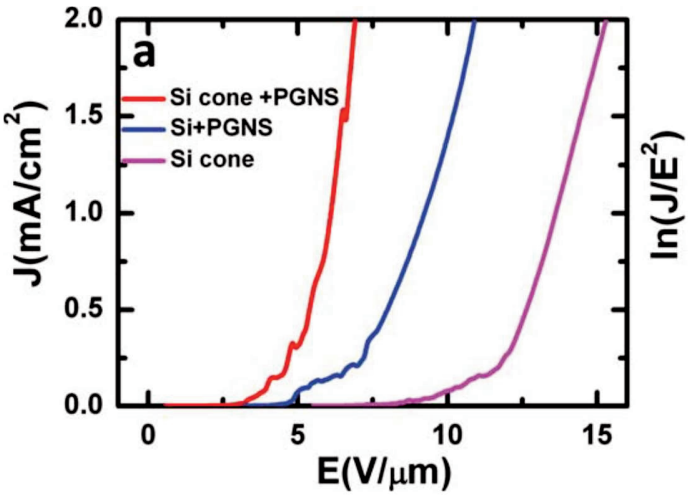


Fig.22 J–E curve showing field emission current density (J) [22]

So, by comparing our obtained sample morphology with their graphene floral structure, it almost most looks similar with even more sharp edges than their sample Hence, we can say that our grown graphene flowers will result into high field emission required for polarization reversal of polymer memory device at low voltage operation

4.3 Raman Spectroscopy

Raman spectroscopy is an integral part of graphene research. It is used to determine the number and orientation of layers, the quality and types of edge, and the effects of perturbations, such as electric and magnetic fields, strain, doping, disorder and functional groups. This, in turn, provides insight into all sp^2-bonded carbon allotropes, because graphene is their fundamental building block. The number of graphene layers (N) in a sample can be determined by elastic light scattering (Rayleigh) spectroscopy, but this approach only works for exfoliated samples on optimized substrates and does not provide

other structural or electronic information. Raman spectroscopy, on the other hand, works for all graphene samples. Moreover, it can identify unwanted by-products, structural damage, functional groups and chemical modifications introduced during the preparation, processing or placement of graphene5. As a result, a Raman spectrum is invaluable for quality control, and for comparing samples used by different research groups. The Raman spectrum of graphite and multilayer graphene consists of two fundamentally different sets of peaks. Those, such as D, G, 2D and so on, present also in SLG, and due to in-plane vibrations, and others, such as the shear (C) modes and the layer-breathing modes (LBMs), due to relative motions of the planes themselves, either perpendicular or parallel to their normal [21].

Figure 24. are the results of raman spectroscopy measurements. From the ratio of G and 2D peaks, we can deduce that the graphene composed of graphene flowers are multi-layers.

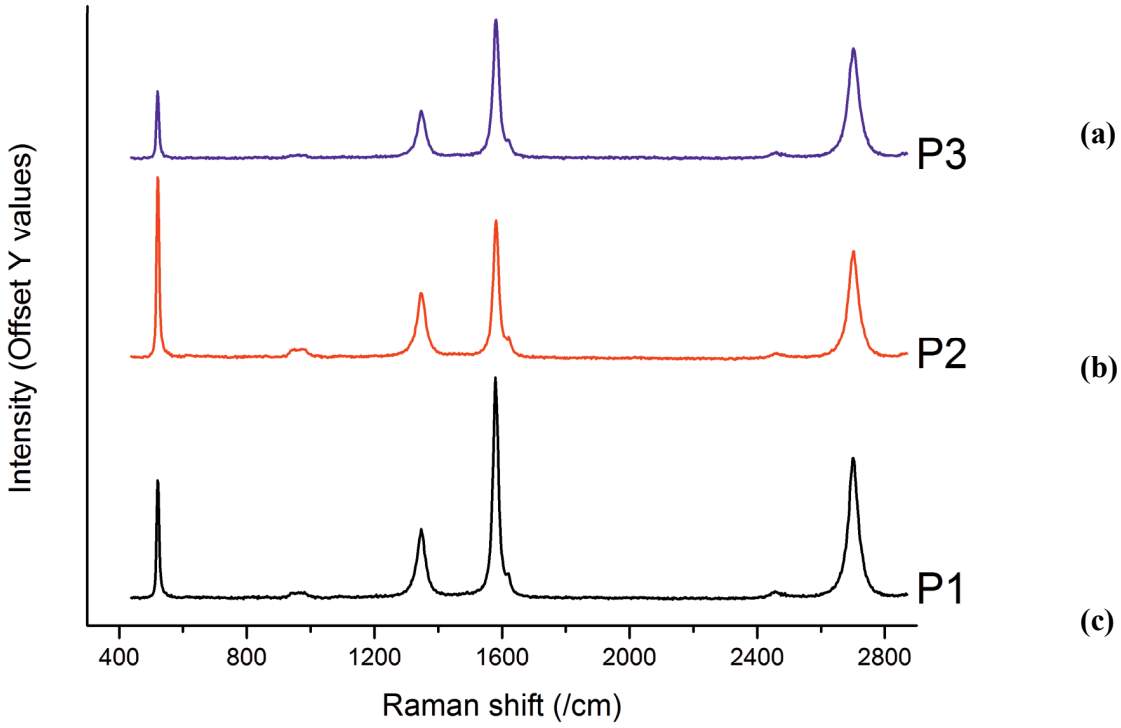


Fig. 23. Raman spectra of grown graphene flowers over substrate

5. Conclusion

In this work, we tried to control the position and distribution of graphene flowers based on many different techniques. Different methods showed different results such as uniformity and density of graphene flower bump, the shape of graphene flower bump. Using this engineering, graphene flower can be applicable for solution-processed device on large area. In addition, morphology of graphene flowers shows its possibility to be used in polymer memory devices

6. Future Works

The low voltage operating MFM capacitor with a P(VDF-TrFE) film at ±12 V was achieved by employing thin poly pyrrole-poly(styrene sulfonate) acid film as interface layers between both bottom and top metal electrodes [12]. A recent work by Naber et al. has also demonstrated a MFM capacitor with a P(VDF-TrFE) layer operating at 10 V when a PEDOT:PSS layer is inserted between bottom electrode and the ferroelectric polymer [11]. The recent work by Ishiwara et al. [15] has demonstrated an MFM capacitor with a 60 nm thick P(VDF-TrFE) film of approximately operating at the voltage as low as 5 V. In future work, we hope that that our prosed device architecture utilizing Nano structured graphene flowers will result into further low voltage operation of MFM capacitor

7. References

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