A THESIS

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Nondestructive Monitoring of Graphene Conductivity using Electrical Impedance Tomography

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Nondestructive Monitoring of Graphene Conductivity using Electrical Impedance Tomography

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A thesis submitted in partial fulfilment of the requirement for the

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I dedicate this work to My Family

& Professor

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Abstract

The graphene film has a good potential structure to be used in the future electronic applications. It has a two-dimensional (2D) structure and also it has many excellent properties. The graphene is synthesized through chemical vapor deposition (CVD) process and then it is transferred to the other substrate surfaces. The different surfaces can create various properties of samples, which can lead to variation in its electronic structure, morphology, etc. Therefore, we need to measure the conductivity of the graphene film after sample fabrication. The commonly used methods for measuring conductivity are 4-point probe method, Van der pauw method, Raman spectroscopy, etc. This methods are destructive and only provide information of conductivity measurement across small area. There is a necessity for development of efficient non destructive methods for imaging of whole graphene sample. Electrical resistance tomography (ERT) is a simple, fast and non destructive method that can be used for the monitoring graphene sample. The ERT is an imaging modality in which the internal conductivity distribution is reconstructed based on the measuring voltage on the object surface. In this study, we present a simple method for large sample conductivity measurement of a graphene using ERT. The application of the ERT method has no limitation to the size of the graphene film for conductivity measurement. Electrical conductivity measurement of large size sample is possible and also defects can be investigated by using ERT method.

The conductivity profile results with ERT are discussed, showing the potential of electrical resistance tomography as a reliable and accurate nondestructive technique for the electrical conductivity measurement of graphene sample.

Keywords: Graphene; Electronic Structure; Nonhomogeneous; Electrical Impedance Tomography; Electrical Resistance Tomography; Conductivity; Boundary Estimation; Inverse Problem; Forward Problem.

Abbreviations

EIT	Electrical Impedance Tomography
ERT	Electrical Resistance Tomography
ET	Electrical Tomography
CVD	Chemical Vapor Deposition
2D	Two Dimensional
ECT	Electrical Capacitance Tomography
LCR	Inductance Capacitance Resistance
FEM	Finite Element Methode
PMMA	Poly methyl methacrylate
APS	Ammonium persulfate

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1. Introduction

Electrical tomography (ET) method has been developed as an alternative method to the available medical imaging techniques, for example computed tomography(CT) scan, magnetic resonance imaging, X-ray imaging and other purposes[1]. ET has more advantages in applications related to the imaging in the medical field. Normally, ET methods can be divided based on the different categories according to the electrical quantity to be measured. It can measure magnetic permeability, electric permeability and electrical conductivity which belongs to EMIT, ECT, ERT respectively. We can measure electrical conductivity of any 2D material for electronic application. The graphene film has a good potential structure for the future in the electronics. It has a two-dimensional(2D) structure with many excellent properties. The graphene is synthesized through chemical vapor deposition (CVD) process and transferred to the other surfaces. Electrical conductivity is a material's capability to conduct electricity. It is represented by sigma (σ). It can be calculated by measuring the resistivity and this result will determine how well a material counter the flow of the electric current. After sample fabrication the conductivity evaluation of the graphene film is required. The excellent conductor has a low resistivity and high conductivity[2]. We can also measure the quality, defect in many ways of the sample. The measurement of electrical properties of graphene film can be assessed with different techniques. Such as, the van der Pauw (vdP) methods, transmission line measurements(TLM), 4-point probe method, photolithography, Raman spectroscopy and many others technique[3]. The above mentioned methods are destructive and effective for the measurement at small area.

However, electronic application of large scale graphene films requires fast and accurate method to measure their own electrical properties. The ERT method is a contact method which can finding for conductivity distribution of graphene sample by placing electrodes on the boundary of graphene sample. The conductivity measurements are performed on samples from boundary sides. Also, ERT method is available for many different applications[3]. In the following, two strategies, adjacent and opposite techniques are performed by injecting current at two pair of electrodes and measuring of voltage at pair of other all electrodes.

Here, the ERT is an imaging technique in which the internal conductivity distribution is reconstructed based on the measured voltage on the object surface[4]. The electrical conductivity measurement of large size sample is possible and also defects can be investigated by using electrical resistance tomography method[5]. This technique is not bound to the sample size. The electrical resistance tomography can be used as a reliable, fast, non-destructive and accurate technique for the electrical conductivity measurement of graphene sample.

1.1 Scope of the Thesis

The scope of my thesis is about a nondestructive method for determining the conductivity profile of the large graphene sample. The graphene has been used for many applications. The graphene and other 2D materials have a better electrical properties to use for future electronic device. However, 2D materials have different properties. In this study, the conductivity measurement is performed in large area CVD synthesized sample by using electrical resistance tomography. The graphene is used for thin films, photonic circuits, solar cell, medical sector, industrial, fast charging battery, super capacitor and others many sectors. So, measurement technique needs to be accurate, fast and can able to determine properties of an entire sample area for potential application. But the method needs to be non-destructive without damaging the sample with less measurement time. Electrical resistance tomography is a kind of nondestructive method that uses electrodes at the boundary of graphene sample and small alternating currents are passed through these electrodes and resulting voltages across the electrodes are measured. With ERT the conductivity is determined across whole sample the defect can be investigated.

2. Literature Review

This chapter starts with a brief overview of conductivity measurement technique and also discusses about different methods used for measurement of graphene sample. Graphene is an ideal material for future research. It is very hard to achieve defect-free graphene[6]. There have many synthesis technologies of graphene. For example, mechanical, chemical and others synthesis technologies have available to make a graphene sample.

Why conductivity measurement is required in the graphene sample?

The conductivity measurement is very important part after synthesis of graphene. It is opposite of electrical resistance. The high electrical conductivity is the a main characteristic feature of graphene. The measurement of graphene is needed for quality control, conductivity measurement and defect identification also.

The current-voltage characteristics using ERT can be used to determine the conductivity of graphene sample. From resistivity, we can estimate conductivity. There have many ways to determine conductivity. There are several techniques that have been applied for measuring conductivity of graphene.

1. 4-point probe method

2. TLM (transmission line measurements)

3. THz-TDS (terahertz time-domain spectroscopy)

4. vdP- van der Pauw

5. Raman Spectroscopy and others

The four-point probe method is developed for measuring electronic properties of graphene. This method is fast and simple to use. The four electrodes positioned are applied to square pattern measurements of graphene[8], that shows in Fig 1.

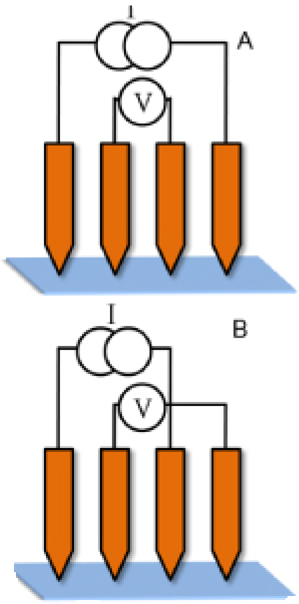

Fig 1. 4-point probe method

Here, outer electrodes are used for injecting current and other inner electrodes are used to measure the corresponding voltage drop. Without contact on graphene surface, it is very difficult to measure the conductivity. So it is a contact measurement technique and destructive method.

To measure over the whole graphene surface, it needs several multiple measurements hence the measurement time is high. THz-TDS method in transmission geometry has been applied in order to measure, accurate mapping of graphene conductivity and mobility over large area. Graphene conductivity is determined within short time by using terahertz transmitted through the graphene film[7]. But there is a limitation for this method like, network set-up would be more complicated to use this method and also time dependent THz pulse spectrum analysis.

3. About Graphene

3.1 What is Graphene

Graphene is a single layer of carbon atoms in a two dimensional (2D) honeycomb structure. This structure of sample is shown in Fig-2. The carbon atom is sp2 orbits structure[6] hybridized in graphene with a C-C bond distance end to end of 0.142 nm. It is first indeed 2D crystalline material. It is able to see as a basic structural building block. The graphene forms graphite with a constant inter level distance of 0.335 nm. Graphene has been discovered in 2004[9], lots of researcher has been focused on this sensational material. The graphene has very good electronic properties, therefore have potential for developing future devices. The graphene is an excellent electronic conductor.

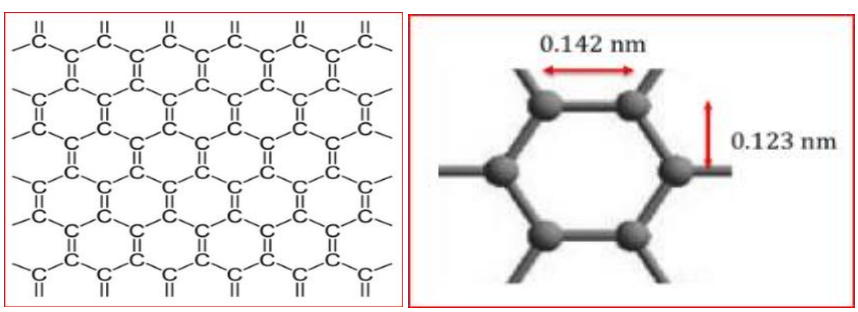

Fig 2. Graphene structure and shape

The graphene has good electrical charge carrier mobility till ~ 200, 000 cm^2/Vs[10] and the conductivity of graphene is higher than silver metal.

3.2 Application of Graphene

Graphene has lot of potential applications. It is a world thinnest, conductive, strongest material. The graphene is used in industry and research sector around the world. The application of graphene is available for IC, distillation, sensor, transistors, ultra-capacitor, batteries, protective coating, solar cell, super capacitor, flexible mobile, flexible LED screen and many other purposes.

3.3 History of Graphene

The fabrication of graphene was filed in October, 2002, "Nano-scaled Graphene Plates''. After two years, in 2004 Andre Geim and Kostya Novoselov at University of Manchester.

Fig 3. Picture of the Nobel Prize winner in Physics 2010

Andre Geim and Kostya Novoselov also were awarded the 2010 Nobel Prize in Physics "for ground breaking experiments regarding the two-dimensional material graphene"[11].

3.4 Graphene Properties

3.4.1 Chemical Properties

Chemically, graphene is the most reactive form of carbon. Carbon atoms at the border of graphene sheets have distinct chemical reactivity. It can burn at temperatures (e.g, 350 °C). The graphene has very strong bond with each of other carbon atoms. After removal of graphene oxide, there is no further oxidation. Basically, we can do this by using chemical methods[10,12]. The carbon with each single atom is exposed to the chemical effect from two sides.

3.4.2 Mechanical Properties

Graphene is the strongest, thinnest wonderful material. The scientists have discovered a technique called atomic force microscopy to estimate graphene strength. Where they found it is harder than diamond and 300 times stronger than steel. The extensible strength of graphene exceeds 1 TPa. It is stretchable up to 20% of its primary length[12].

3.4.3 Electronic Properties

Graphene is a zero-overlap semi-metal. It also has very high electrical conductivity[13]. The electrons are able to flow through graphene more easily better than copper. Graphene has high charge carrier mobility standards of 200,000 cm^2/Vs at low temperature. The resistivity of graphene is approximately 10^{-6} Ωcm.

3.4.4 Thermal & Optical Properties

The thermal conductivity of graphene–5000 W/m.K (Diamond has 1200 W/m.K). It is transparent in nature 98% visual transmission rate. Graphene is a thinnest material with thickness 0.335 nm[14].

3.5 Synthesis Technology of Graphene

Mainly, there are 3 main ways to synthesize graphene, that are shown in Fig 4.

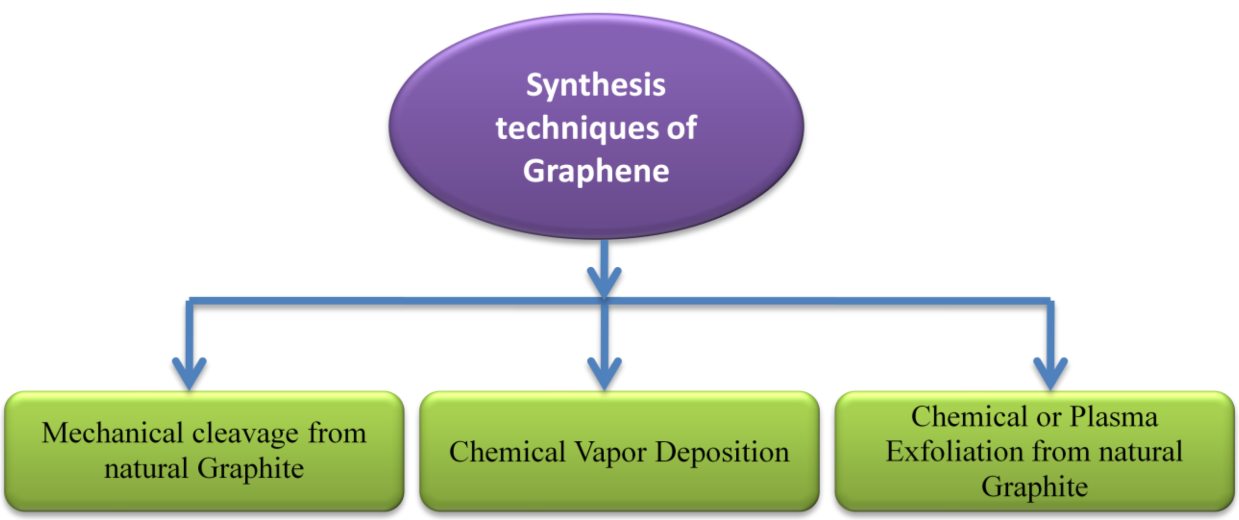

Fig 4. Schematic diagram of the synthesis technique of the graphene

Mechanical cleavage from the natural graphite method is used to synthesize small number of layers. This method has been used for the first time to isolate graphene from graphite by using sticky tape. This mechanical method can be used for high quality sheets of graphene sample. But this is also a slow method. CVD is a great technique to synthesize large-area less defective graphene[15].

The common characteristics that may include high homogeneity normally, high quality, fine grains, low cost, good control over layer number and more. In a chemical exfoliation process, graphite is exfoliated by large ions among the graphite layers.

The chemical synthesis is the same procedure which consists of the separation of graphite oxide, dispersed in a solution, followed by reduction[16].

3.6 Sample Preparation of Graphene

If graphene were as thick as a piece of paper. A piece of paper would be as thick as a two-storey building. Many single atomic layers are available in graphite. We need to exfoliate from them. But the atomic layers are weakly bonded among them. So to exfoliate them scotch tape is used, that can separate some of layers[17]. This method is applied for the first time for synthesis of 2D materials and made 2D materials ever. But this method is not a proper method for making a monolayer graphene film. It is a not commercial and industrial way to make graphene. Chemical methods have come to help in the fabrication process. CVD method is a chemical method, which can grow a thin film of graphene[18].

4. Introduction to ERT

Electronic application of large-area graphene films requires fast and accurate methods to measure their electrical properties. ERT method is a technique that can offer fast and non-destructive measurements. The conductivity distribution can be determined on a large area graphene sample by using electrical resistance tomography. This technique is not bound to the sample size.

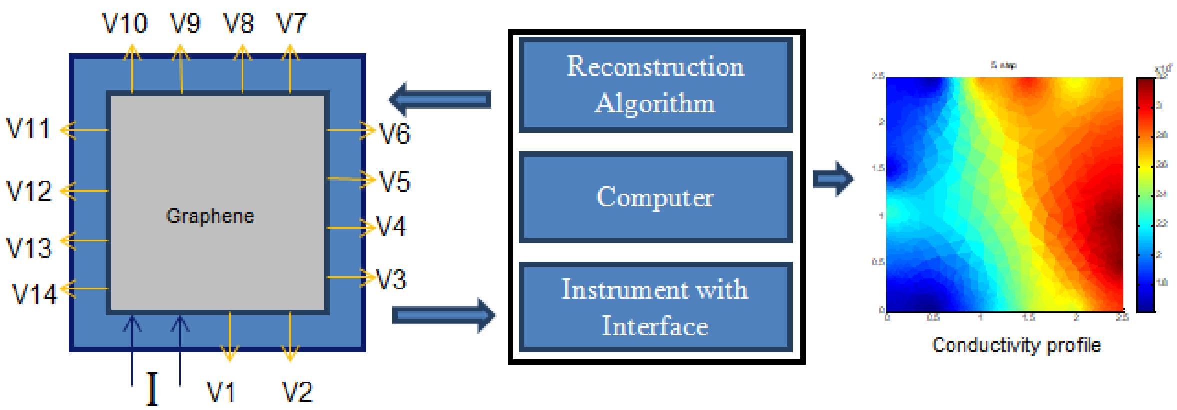

Fig 5. Schematic diagram of electrical resistance tomography

ERT is an imaging technique in which the interior conductivity distribution is reconstructed based on the measured voltage on the object surface[20]. It is an imaging method applied to determine the surface resistivity distribution using the measurements on the boundary of graphene sample. The measurements are recorded from the 16 electrodes attached to the boundary of the sample.

Image reconstruction in EIT is done using forward and inverse problem. Forward problem is about the computation of boundary voltages using distribution of conductivity and injected currents (Fig 6a). In inverse problem the conductivity distribution on graphene surface is estimated from measured voltages and injected currents (Fig 6b).

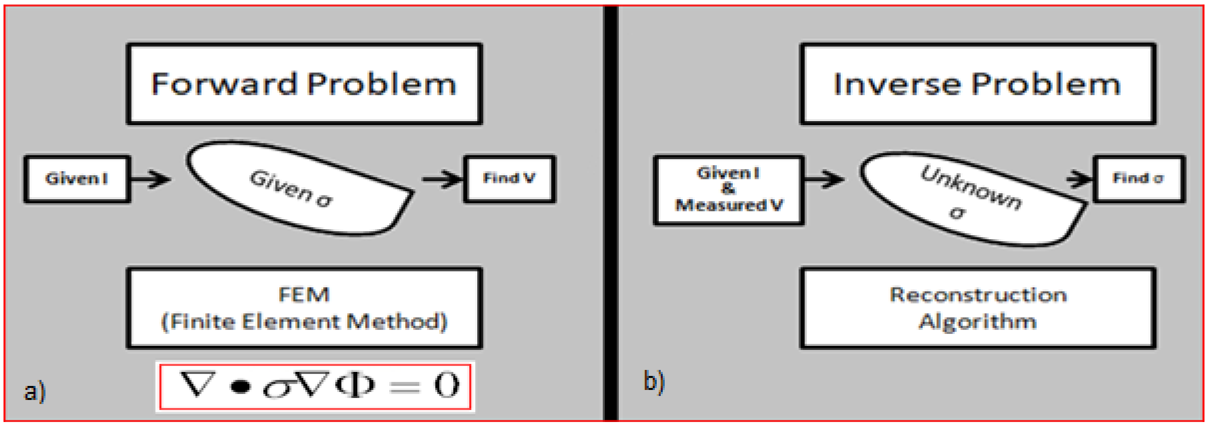

Fig 6. Forward vs. inverse problem in ERT

4.1 Forward Problem

The forward problem is used to calculate the boundary voltage from known conductivity distribution and applied currents[22]. For the modelling and simulation purposes, the mathematical model is established in electrical resistance tomography system for forward problem and inverse problem[21]. In ERT, current is applied to the sample surface through the electrodes and voltage are measured across the electrodes. The relation between electrical potential u in Ω and resistivity distribution (ρ) are governed by partial differential equation derived from Maxwells equation.

$$\nabla \bullet \frac{1}{\rho} \nabla u = 0 \quad \text{in } \Omega \quad (1)$$

$$u + z_\ell \frac{1}{\rho} \frac{\partial u}{\partial v} = U_\ell \quad \text{on } e_\ell,\ \ell = 1, 2, ..., L \quad (2)$$

$$\int_{e_\ell} \frac{1}{\rho} \frac{\partial u}{\partial v} dS = I_\ell \quad \text{on } e_\ell,\ \ell = 1, 2, ..., L \quad (3)$$

$$\frac{1}{\rho} \frac{\partial u}{\partial v} = 0 \quad \text{on } \partial\Omega / \bigcup_{\ell=1}^{L} e_\ell \quad (4)$$

Where, boundary voltage=U_ℓ, v is the outward unit normal, number of electrode=L, and z_ℓ is denote the contact impedance [23]. Also u electric potential. Here, I_ℓ =current, $e_\ell =$ electrode, $\partial\Omega$ = surface and resistivity distribution are ρ .

Using governing equation (1) and boundary condition (2-4) for a given conductivity, the boundary voltage can be determined analytically for homogeneous conditions. If the conductivity is not homogeneous, it is difficult to determine the solution analytically. Therefore, often numerical methods are used to compute the boundary voltages.

4.2 Data Acquisition

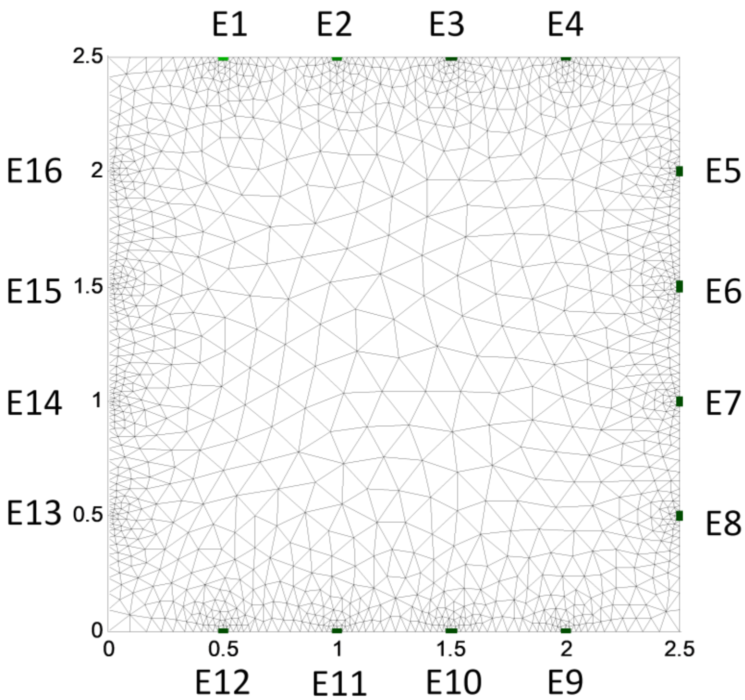

Fig 7. Adjacent and opposite method use for injection current

The sixteen electrodes are connected to the boundary of the graphene sample as shown in Fig 7. The electrical currents are supplied across the electrodes and the voltages are measured.

Adjacent method: current is injected through (E1 and E2) electrodes and measured the voltage from all remaining parts. After that current port is changed to the other neighbouring electrodes (E2 and E3) and measure the voltage again from across to all other electrodes[24].

Opposite method: The current is injected through opposite electrodes, i.e. (E1 and E9) and the voltage is measured from all remaining electrodes (E3-E4, E4-E5, E5-E6….) after that in the next current pattern new pair of electrodes are chosen for instance (E2 and E10) and the voltage is measured again from across to all other electrodes.

4.3 Finite Element Method (FEM)

The finite element (FE) method is used to obtain the mathematical solution of the governing equation. The domain is divided into many finite number of small triangular elements and it is assumed that the conductivity is constant within each element. The forward problem can be formulated as a system of linear equations, AB= f where $A \in \mathbb{R}^{(N_n+L-1)\times(N_n+L-1)}$ denotes the system matrix, $B \in \mathbb{R}^{(N_n+L-1)\times 1}$ is the forward solution, $f \in \mathbb{R}^{(N_n+L-1)\times 1}$ is the data vector that is a function of the injected currents, and N_n is the number of nodes in the FE mesh. Details of the ERT forward problem using the FE method can be found in[25].

4.4 Iterative Gauss–Newton method

For the inverse problem, Gauss-Newton method is used as a solver of the inverse problem. That can be estimate interior resistivity distribution of the domain. Because of non-linear ill-posed problem, the objective function, including regularization and previous information is formulated to reduce the ill-posehness[23,26].

$$\Phi(\rho) = \frac{1}{2}\left\{\| U(\rho) - V \|^2 + \alpha \| R(\rho - \rho^*) \|^2\right\} \tag{5}$$

Where, boundary voltage= $U(\rho)$, the actual voltage measured of boundary across the electrode=V, regularization parameter=R, Denotes prior fact= $\rho*$

$$\hat{\rho}_i = \hat{\rho}_{i-1} + \left[J_{i-1}^T J_{i-1} + \alpha R^T R\right]^{-1} \left[J_{i-1}^T \left(V - U_{i-1}\right) - \alpha R^T R\left(\hat{\rho}_{i-1} - \rho^*\right)\right] \tag{6}$$

$$\hat{\rho}_i = \hat{\rho}_{i-1} + \left[J_{i-1}^T J_{i-1} + \alpha R^T R\right]^{-1} J_{i-1}^T \left(V - U_{i-1}\right) \tag{7}$$

5. Conductivity Estimation of Graphene using ERT

In this part, the numerical result is presented for conductivity estimation of graphene sample by using ERT. The reconstruction results for the proposed method with simulated data are given in Fig 8. The sample size, length was (2.5 × 2.5) cm^2 and 16 electrodes is placed on the boundary. Electrodes are placed at .5 cm apart of each side of the square sample. The shape and size of mesh for numerical simulations is same with experiment sample.

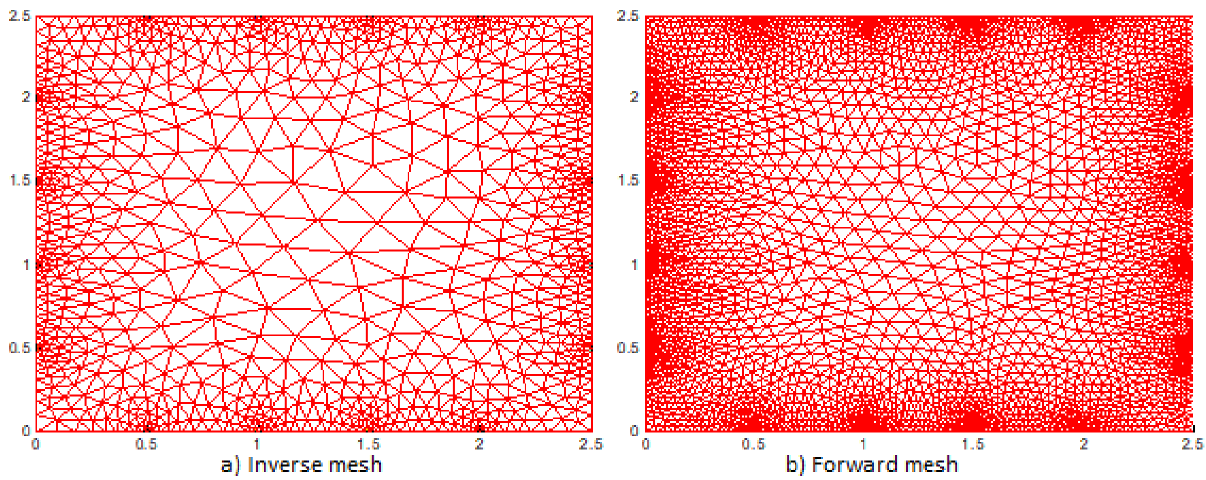

Fig 8. Electrode configurations and mesh structure

Here, two different meshes are used, fine mesh is used to generate true data and coarse mesh is used to estimate the conductivity distribution. Two different meshes are used here to avoid inverse crime[27]. The FE mesh with 6700 triangular elements and 3560 nodes shown in (Fig 8b) is used in the forward problem to generate the true boundary voltages. Inverse mesh nodes with 943 and 1675 triangular elements is used for estimating the resistivity distribution (Fig 8a).

A small alternating current of 1 mA are applied across the electrodes and the resulting boundary voltage are measured. Numerical experiments are performed with single and multiple targets, having a shape like a circular shaped defect and two long scratches. The conductivity of the background is set to 6.7×10^4 S/cm and defect which is target has a conductivity of 0.0001 S/cm. The numerical experiments are measured with single and multiple targets, having two types of shape like a round circular shape and another one are too long scratches[28].

5.1 Numerical results with Case-1

The regularization parameter is considered as 1×10^{-5} during image reconstruction. For noise measurement 1% relative white Gaussian noise was added to the generated voltage to depict real measurements. The true image is shown in (Fig 9a) and also reconstruction image is shown in (Fig 9b). As seen from Fig 9, it can be noticed that defect is estimated with good accuracy. Moreover, the reconstructed background conductivity value is close to the true conductivity value. The colour map represents the range of conductivity value across the sample. Here, red colour resembles relatively high conductivity and the blue colour resemble relatively low conductivity.

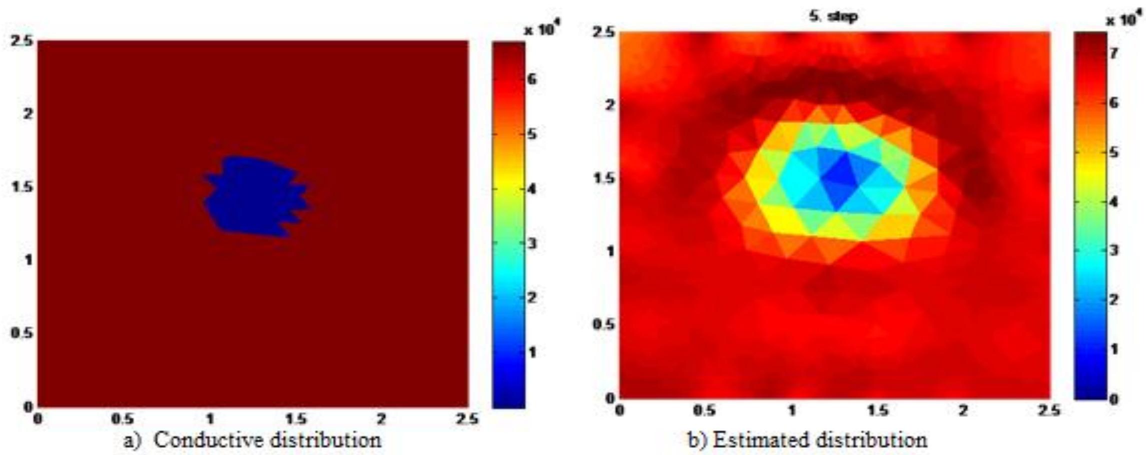

Fig 9. Conductive estimated in ERT for numerical simulation with circular (case-1)

5.2 Numerical results with Case-2

In case 2 with two scratches on sample surface, the regularization parameter was set to 1×10^{-8}. For noise measurement 0.5% relative white Gaussian noise is added to the true generated voltages. The true image is shown in (Fig 10a) and reconstruction image is shown in (Fig 10b).

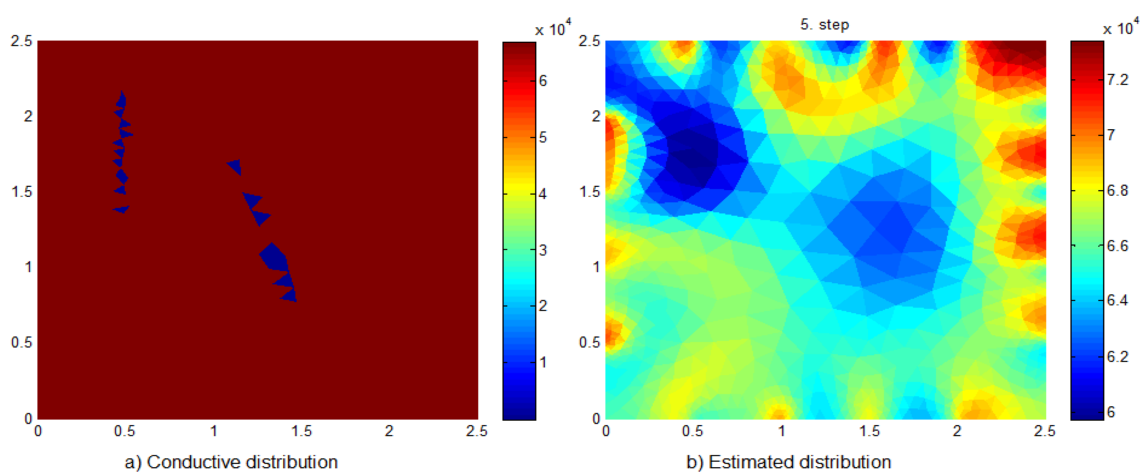

Fig 10. Conductive estimated in ERT for numerical simulation with two long scratches (case-2)

The conductivity of background was 6.7×10^4 S/cm and target conductivity is set to 0.0001 S/cm and amplitude of current was same as previously shown case. The colour scale represent the conductivity value across the graphene sample. Red colour shows as high conductivity and the blue colour shows low conductivity. From (Fig 10) the results for multiple defects are shown and it can be seen that the defects are estimated in the reconstruction image.

6. Experimental Studies

6.1 Sample Preparation

For the single layer graphene sample is grown over 18μm-thick copper foil, methane and hydrogen gas is passed through the vacuum chamber and also high temperature supply is provided. Before the growth of graphene, hydrogen and argon gas with 1000°C is supplied for about 15 minutes for removing the copper oxide. When molecules of methane hit to the copper surface, then the carbon atom get entrap inside onto copper surface and hydrogen atoms could be moved around. This carbon can be deposited as a single layer on copper surface.

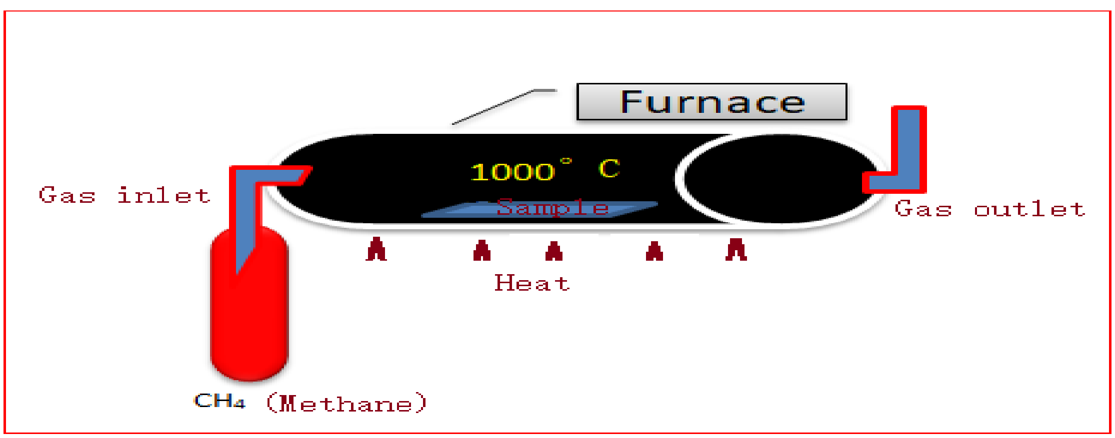

Fig 11. Schematic diagram of tube furnace with reaction system for the synthesis of graphene by CVD

The deposited layer can be various centimetre long and tightly bonded on copper surface. The layer should be transferred to target surface. So organic polymer, poly (methyl methacrylate) can be used by spin-coating on the carbon surface. After that, copper foil was dropped on Ammonium persulfate (APS) to chemically remove the copper layer. After 15 min, the copper will be removed properly. Then PMMA should be washed with DI water to remove the APS clearly and then we can use Si/SiO_2 wafer to put under the graphene sample. Finally, the graphene sample is ready to use for experimental purpose and PMMA is required to remove by using acetone or other solvents[19].

6.2 Experimental Condition

The Agilent 4284A LCR meter is used as an energy source to inject alternating current. A magnitude of 1 mA current with 1 kHz frequency is injected through the electrode attached to the graphene boundary and NI PXI-1042Q is used to measure resulting boundary voltages of the sample. Here, we have used LCR meter for current injection, but there are some parameters to choose. In some cases, choosing the wrong settings can result in poor measurement accuracy.

7. Experimental Result

7.1 Conductivity Estimation with Experimental Studies with Adjacent Method

The current is applied to the sample through the electrodes coated with silver paste on graphene sample and estimated the boundary voltages with adjacent mechanism. The measured voltages using the adjacent method are shown in Fig 12. The magnitude of voltages is not uniform as it can be seen from Fig 12.

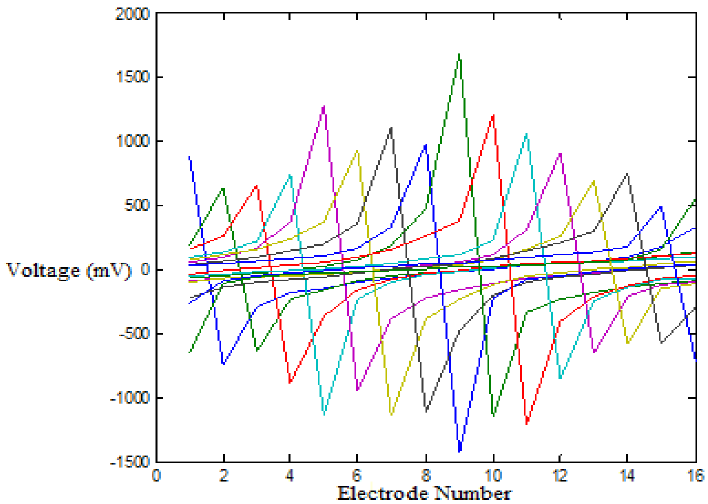

Fig 12. Boundary voltage measured on an experimental Graphene sample with adjacent method

The electrical conductivity measurement of the CVD graphene samples with ERT method is shown in Fig 13. Near electrode-9 in the reconstructed image, the conductivity is low as compared to other area of graphene sample.

The electrical properties of the CVD large size graphene sample were measured by using ERT method. The ERT method used to calculate of the local conductivity value of the whole graphene sample. In Fig 13 shows, conductivity distribution across the graphene sample using the adjacent method. Here, red colour shows high conductivity 4×10^4 S/cm and the blue colour is shows low conductivity of 1.5×10^4 S/cm.

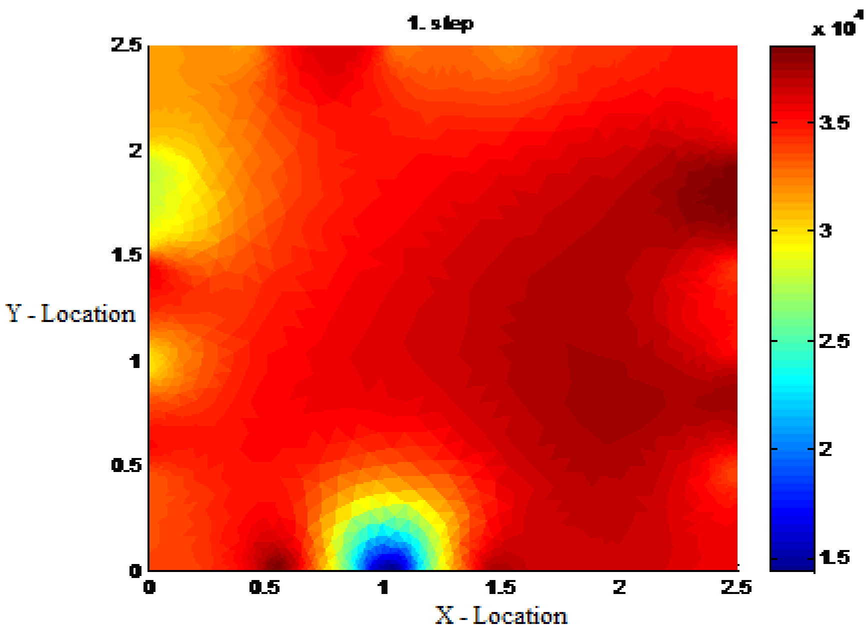

Fig 13. Final conductivity profile of the flow domain with adjacent method

7.2 Conductivity Estimation with Experimental Studies with Opposite Method

Here also, same amount of injected current is supplied to the electrodes of (E1 and E9) then (E2 and E10,……...E8 and E16) and estimated the boundary voltage with opposite mechanism. The corresponding voltage measurements with opposite method are shown in Fig 14.

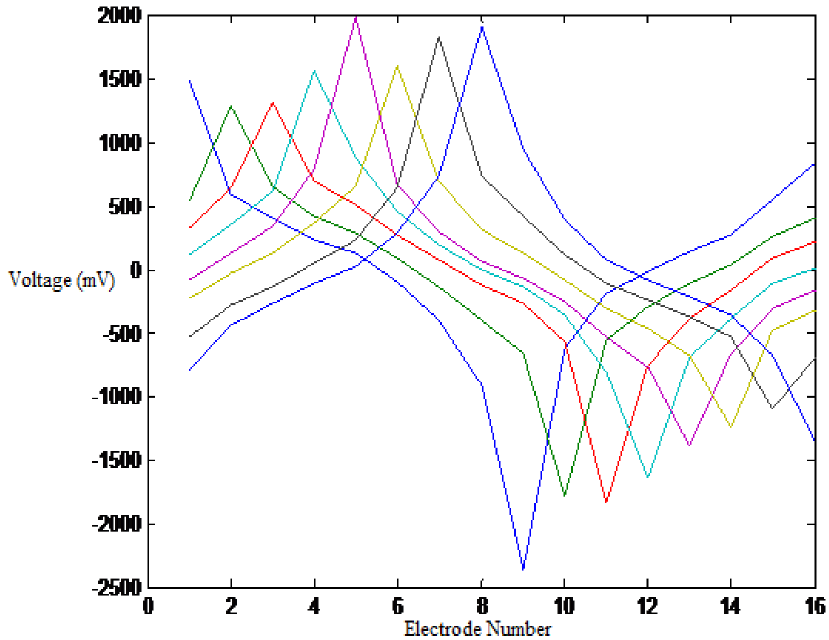

Fig 14. Boundary voltages measured on experimental Graphene sample with opposite method

This ERT method was used to measure the conductivity value of the whole graphene sample with opposite injected current. The Fig 15 shows, the conductivity distribution of graphene sample by using opposite method injection. Here, red colour shows about high conductivity 3.4× 10^4 S/cm and the blue colour is shows low conductivity on an average of 1.2× 10^4 S/cm.

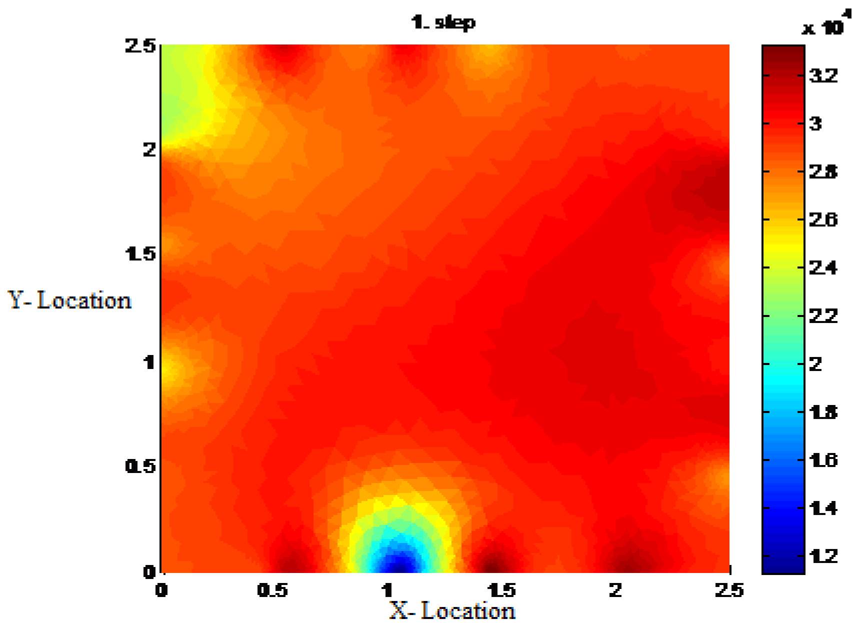

Fig 15. Final conductivity profile of the flow domain with opposite method

Conductivity image pixels have a colour scale for understanding conductivity distribution of the large area graphene sample. The ERT image of the conductivity distribution can be helpful in identifying the regions of low and the high conductivity area. Therefore, this conductivity image can be used to locate the defects present in the sample.

8. Future Works

The importance of this scientific research is about new perspective view on the conductivity measure by using ERT method. In future we can develop conductivity measurement by using ERT and also can demostrate efficient algorithm for improving spatial resolution.

For future work, more research should be conducted such as a graphene conductivity measurement of a large size sample without damage, the effect of contact resistance removal etc. If they have many connections between metal to semiconductor for the interface, there is a possibility for increase of contact resistance. So we should reduce contact resistance, hence the conductivity information from ERT can be considered as the true conductivity of graphene sample.

Further research can provide more effective results with large area sample of graphene. Develop the conductivity measurement technique for any 2D materials. The unique properties of graphene conductivity are investigated by using many methods. But we need to find out the efficient numerical algorithm to determine the conductivity distribution with good spatial resolution.

9. Conclusions

In this study, electrical resistance tomography was used to determine the conductivity of the large graphene sample. Here, we used adjacent current and opposite current injections strategies[29]. We have measured the conductivity distribution of graphene by using ERT method. ERT offers high potential characteristics to image the surface of graphene nondestructivity. ERT is a low cost portable system with fast measurement time, moreover it is not dependent on the sample size.

10. References

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