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A Brief Overview for Fundamental Electrical Characterization Techniques for Thin Films and Nanostructures

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Thin films and nano materials are crucial for the advancement of technology and industrial applications. Electrical characterization of such materials is crucial for understanding the internal characteristics of the materials and to produced advanced and innovative materials. Thin films are special materials which were applied as a layer on a certain surface. Such films were often applied in nanoscale and therefore, they were associated with nanotechnology and nanomaterials. Thin films were used in electronical devices, solar cells, sensors, solar harvesting devices, detector technologies, etc. Electrical characterization of thin films and nanostructures enables researchers to investigate different properties such as impedance, resistance, capacitance under various conditions such as temperature, frequency, voltage, etc. Materials in desired electrical properties could be developed. Similarly, nanomaterials also exhibit unique characteristics where electrical characterization techniques has important role for understanding the intrinsic properties the materials. Nanomaterials such as carbon nanotubes, graphene, and metal nanoparticles exhibit outstanding electrical and electronical characteristics. Electrical characterization of such materials may find applications in energy storage applications, biomedical applications etc. In this report, we briefly discussed electrical characterization methods used for thin films and nanomaterials such as Hall Effect, Four Point Probe Technique, Van der Pauw Technique, Capacitance – Voltage (C-V) Measurements, Electrical Impedance Spectroscopy.

Keywords: *Hall Effect, Four Point Probe Technique, Van der Pauw Technique, Capacitance – Voltage (C-V) Measurements, Electrical Impedance Spectroscopy*

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1. Introduction

Nanostructures are assemblies of atoms and molecules that unite to create distinct configurations spanning from just a few atoms to several hundred thousand atoms [1]. Generally, these formations commence at sizes within the range of a few nanometers and are anticipated to be smaller than 1 micron [2]. These nanostructures fall into various categories, encompassing zero-dimensional structures, exemplified by dot structures; one-dimensional structures, such as nanotubes and nanowires; two-dimensional structures like nano films; and three-dimensional structures, including multilayer films. The intricate and precisely engineered nature of nanostructures opens up avenues for diverse applications, showcasing their potential across a spectrum of scientific and technological domains [3]–[6]. Alteration in shapes, size or forms of nano materials may result in drastic alterations in their internal and external characteristics. Thus, nanomaterials can be applied to electronic technologies, imaging technologies, semiconductor devices, solar cells, sensors, military technologies, telecommunications technologies, and biomedical applications [7]–[10]. Nanorods, nanodots, nanotubes, nanospheres are common forms of nanomaterials which were repeatedly reported in the literature. Thin films are special materials which were applied as a layer on a certain surface. Such films were often applied in nanoscale and therefore, they were associated with nanotechnology and nanomaterials [11]. Different production techniques were reported in the literature some which are co-precipitation, hydrothermal synthesis, thermal vapor deposition or laser vapor deposition and magnetron sputtering etc. [12], [13]. Thin films refer to layers of material with controlled thicknesses, aiming to manipulate structures at the atomic or molecular scale during the manufacturing process. These films are versatile and can be applied to a wide range of surfaces [14]. For scientific, research and development purposes, thin films are often growth on substrates with well-known internal properties for precise characterization. In practical applications, they are generated by depositing a material layer onto a designated surface. The structural characteristics of thin films can vary, resulting from diverse components at the atomic or molecular level. This adaptability makes thin films invaluable in both controlled laboratory settings and real-world scenarios, where their tailored properties find application in numerous fields [15], [16]. Thin film characterization techniques were often applied in scientific researches and industrial applications to obtain deep understanding of intrinsic properties of such materials [15], [16]. Understanding the electrical properties of such materials is crucial for optimizing, reducing, or enhancing specific properties of these materials to meet the needs of the industry. Electrical characterization techniques contribute to the development of high performance materials in a cost effective way. It is also essential for electronic technologies, telecommunications technologies semiconductor devices, solar cells, military technologies and sensors, [12], [17]–[19]. For this purpose, Hall Effect, Four Point Probe Technique, Van der Pauw Technique, Capacitance – Voltage (C-V) Measurements, Electrical Impedance Spectroscopy techniques were often used for electrical characterization.

In our report, we have briefly discussed the Hall Effect, Four Point Probe Technique, Van der Pauw Technique, Capacitance – Voltage (C-V) Measurement, and Electrical Impedance Spectroscopy techniques which are fundamental methods used for the electrical characterization of nanostructures and thin films.

2. Hall Effect

Hall effect measurement is an electrical characterization technique that examines the change in direction of the current passing through a material under the influence of an externally applied magnetic field. The Hall effect is named after the physicist Edwin Hall, who discovered it. It is often used for the characterization of thin films and nanomaterials. Most of the thin film applications focus on semi-conductor based implications. Hall effect measurements are used to determine the conductivity type (n-type or p-type), charge carrier density, and carrier mobility of the material. Such characteristics may help researchers to understand the internal characteristics of the materials where suitable material can be used for required applications. In hall effect measurements measured particle was subjected to a high magnetic field. When linearly moving charges are subjected to a magnetic field perpendicular to the direction of motion, they follow a curved path. This force that changes the flow direction of the charge carriers is known as the Lorentz force. While negatively charged electrons accumulate on one side of the surface, the other side becomes positively charged. Due to this charge distribution, the electric field generated within the material impedes further electron migration. As long as the current flow continues, a constant potential difference is maintained. Figure 1 illustrates the Hall effect generated by the magnetic field.

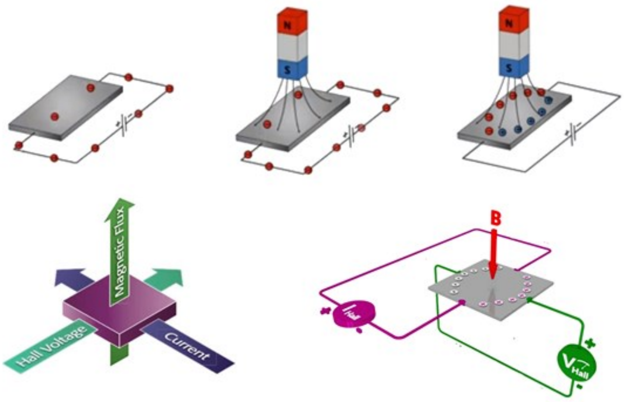

Figure 1: A schematics illustrating the Hall effect measurement.

Hall effect measurements are typically performed with a device called a Hall sensor, which is activated by an external magnetic field. When the magnetic flux density exceeds a pre-set threshold value, the sensor detects it and produces an output voltage called Hall voltage (V_H).
Such voltage is formulized as follows:

$$V_H = \frac{IB/d}{2ne} \qquad \text{Eq.1}$$

where I is current, B magnetic flux, d thickness of the conductive wire, n is the density of the electron transferring charges, e is the charge of an electron.

The Hall effect measurement system can measure the following basic parameters:

(I) Hall Voltage (V_H): Represents the potential difference created on the Hall probe. This voltage occurs depending on the strength of the magnetic field and the direction of current flow.
(II) Magnetic Field Strength (B): Represents the intensity of the external magnetic field applied to the Hall probe.
(III) Current Density (I) : Represents the intensity of the current passing through the material.

Hall effect measurements are widely used in the electrical characterization of semiconductors, metallic materials, semiconductor devices, and material samples. It is particularly important for determining the carrier density and mobility of semiconductor materials, optimizing the conductivity properties of materials, or evaluating the performance of semiconductor devices. In nanotechnology studies, it has been determined that graphene, a two-dimensional form of carbon with a single-atom thickness, exhibits quantum Hall effect.

3. Conductivity Measurements

Conductivity measurements are techniques used to determine how well a material conducts electric current and to characterize its electrical conductivity properties. These measurements are important for understanding the material's conductivity level, resistance value, and electrical characteristics. Therefore, materials in desired conductive and resistive properties could be used scientific and industrial applications. Some fundamental conductivity measurement techniques are as follows:

3.1. Four Point Probe Technique

The four-point probe technique is a method used to measure the resistance properties of a sample; the technique is particularly preferred for accurate measurements of low-resistance materials. In the method, four separate probes are placed on the sample, where two probes send the current, and the other two probes measure the current. By passing the current through the two outer probes and measuring the voltage across the inner probes, the sheet resistance of thin films is able to be calculated. This arrangement allows measurements to be made without being affected by probe contact resistance where enabling precise resistance measurements can be performed. Hence, the material resistance is calculated, and the conductivity value is determined. Surface resistance measurements are primarily used in the production and quality control of conductive and semiconductive coatings, materials with thin film coatings, OLEDs, and glass materials, including ceramics, textiles, and nanomaterials. While scanning the surface with a four-point probe, the voltage-current ratio is measured for each step. Therefore, surface mapping can be obtained. Such data then can be used to characterize the conductive and resistive properties of the measured materials.

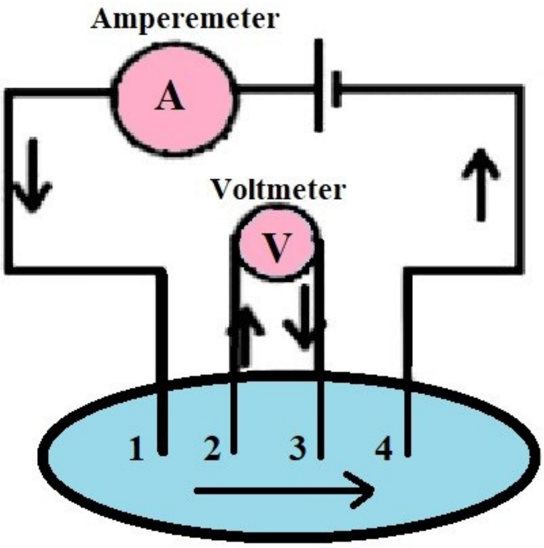

Figure 2: The schematics illustrates four point probe technique.

3.2. Van der Pauw Technique

The Van der Pauw method is a technique used for conductivity measurements in irregularly shaped or nearly two-dimensional samples. It is employed to map the conductivity distribution across the surface of a sample. In this method, a current is applied around the perimeter of the sample, and voltage is measured. Four contact points are placed along the perimeter of the sample, with two points applying the current and the other two points measuring the voltage. Subsequently, the positions of the four points are changed in a clockwise direction, and the measurements are repeated. Using this method, the material's resistivity and conductivity value are calculated. By applying a magnetic field and measuring the corresponding change in diagonal Van der Pauw resistance, the Hall coefficient of the sample can also be determined. To perform a reliable measurement, the sample thickness should be small compared to the distance between the contact points. Also, no holes, punctures or tears should be seen in the sample where shape should be mathematically continuous without containing. Be sides no islands made of highly conductive material should be the part of the material. The four contact points should be located along the sample's edge, and they need be small compared to the sample's area.

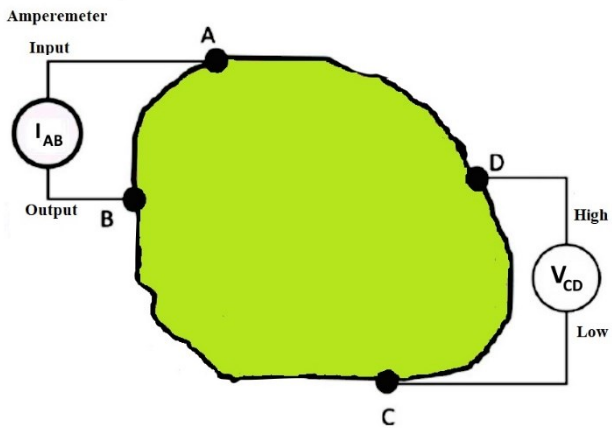

Figure 3: The schematics illustrates Van der Pauw Technique.

4. Capacitance – Voltage (C-V) Measurements

Capacitance-Voltage (C-V) measurement is a technique used to examine and characterize the electrical properties of thin films and nanomaterials. This method determines how the material's capacitance responds to changes in voltage. C-V measurements are widely used in various fields, including the design and characterization of semiconductor devices, semiconductor research, and the production of electronic components.

The basic steps in C-V measurements are as follows:

(I) Sample Preparation: For C-V measurements, the dielectric material is placed on an appropriate substrate, and electrical contacts are established. The substrate provides electrical and mechanical connections for the sample. The sample should be clean. If the sample is not clean a proper cleaning procedure should be applied. Sample should be flawless where any cracks or dents in the sample may alter the capacitance characteristics of the sample.
(II) Flatband Measurement: In the initial step, a voltage is applied to the material to create a flatband condition. In this state, the electrical charges on the material are balanced, and the capacitance value is at its lowest level. This step establishes the reference point for the measurement.
(III) Voltage Sweep: In the voltage sweep step, varying voltage values are applied to the material, and the capacitance is measured at various voltage levels. As the voltage increases or decreases, the material's capacitance changes, allowing the analysis of the material's electrical properties.
(IV) Frequency and Temperature Variation: C-V measurements are often conducted at different frequencies and temperatures. This allows us to examine how the material responds to changes in frequency and temperature. It is quite common for thin films and heterojunctions to observe an altering conductive characteristic for altering frequencies.

The data obtained from C-V measurements are represented as voltage-capacitance graphs. These graphs provide information about the material's capacitance-voltage characteristics and other electrical properties. For example, information such as the material's dielectric constant, effective carrier density, surface state, and charges at interfaces can be determined through C-V measurements.

5. Electrical Impedance Spectroscopy

Impedance is the resistance that circuit elements exhibit to alternating current as a function of frequency. Electrochemical Impedance Spectroscopy (EIS) provides comprehensive information over a wide frequency range by measuring the impedance (Z) of a material as a function of frequency. This technique helps to determine the electrical properties and interactions of the material. The impedance value is calculated for each frequency using the measured current and potential values, resulting in a complex number with real and imaginary parts.
Electrochemical impedance is often determined by applying an AC potential to an electrochemical cell and measuring the current passing through the cell. The interaction between the electrodes of the electrochemical cell is represented by the Randles cell (RC circuit).
The working electrode is the electrode under analysis. The signal generator applies small-amplitude potential signals (AC signals) to the working electrode within a specific frequency range, and current flows through the material. In this case, the impedance developed on the material is the ratio between the voltage from the signal generator and the current. Potential changes on the working electrode are determined relative to the reference electrode. The counter electrode is the electrode that either provides or collects current.
The Randles cell includes components such as electrolyte resistance arising from the electrolyte solution, surface resistance resulting from electrochemical reactions occurring at the electrode/electrolyte interface, and surface capacitance formed by the accumulation and distribution of ions at the electrode/electrolyte interface.
Nyquist plots are commonly used for impedance data evaluation. The Bode plot is another graph used for finding impedance (Z) and double-layer capacitance (C_{DL}). These graphs visualize the frequency-dependent impedance changes of the material. The value of double-layer capacitance depends on factors such as electrode potential, temperature, ion concentrations, ion types, and oxide layer.

An electrochemical cell can be modelled as an electronic circuit which as can be shown in Figure 4.

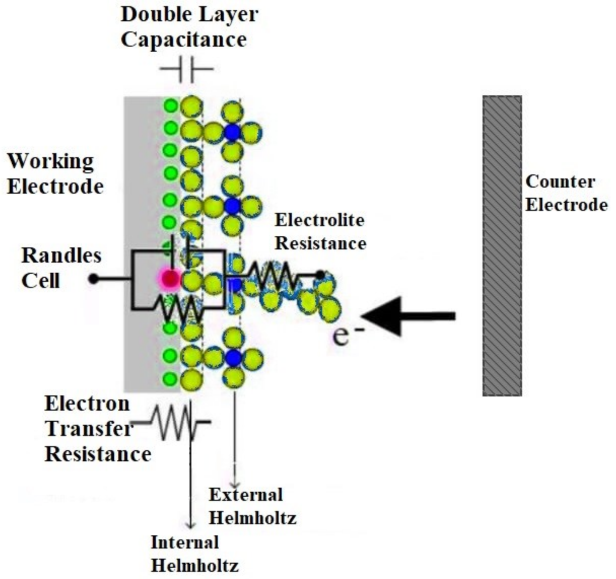

Figure 4: The schematics illustrates electrical impedance spectroscopy.

EIS (Electrochemical Impedance Spectroscopy) is widely used in various fields, including the characterization of material surfaces, investigation of electrochemical interactions, evaluation of the performance of electrochemical cells, design of sensors, and biomedical applications. Additionally, it serves as a crucial tool for the characterization of electrochemical interfaces and reactions in electrochemical energy storage devices such as batteries, fuel cells, and supercapacitors. EIS finds extensive application in diverse fields, particularly in electrochemistry, materials science, electrochemical energy storage systems, biology, and medicine.

6. Conclusion

The electrical characteristics of the materials has a crucial role for thin films and nanomaterials which can find application in different fields such as semi-conductors, solar harvesting devices, photocells, photodetectors, etc. In our report, we provide a brief information about electrical characterization techniques which were commonly used for thin films and nanomaterials.

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Author Contributions

Erdal KARAKUŞ: Conceptualization, writing,
Mumin Mehmet KOC: Editing, English check
Burhan COŞKUN: Conceptualization, writing, supervising

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