Chapter 9 Obtaining Process of Zinc Oxide (ZnO) in laboratory with Evaporation and Thermal Oxidation

Capítulo 9 Proceso de Obtención de material Óxido de Zinc (ZnO) en laboratorio con Evaporación y Oxidación Térmica

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Abstract

The following project shows the results of experiments related to obtaining zinc (Zn) nanoparticles on glass substrates by the thermal evaporation process in a high-temperature tubular furnace at 800 ° C, with a flow of 400 sccm of inert gas (Argon) used as carrier gas, to deposit metallic Zn particles in the cold zone of the furnace, likewise the metallic Zn deposit samples were thermally treated with the intention of being used as a proposed n-type semiconductor material based on metallic oxides, which was formed through the thermal oxidation technique in the same high-temperature tubular furnace at 500°C, with air flow at 600 sccm to form an oxidizing environment and consequently form continuous layers of material Zinc Oxide (ZnO) and be analyzed morphologically with Scanning Electron Microscopy, structurally with X-ray Diffraction and electrically by placing metallic contacts. s to the samples to measure their Current-Voltage curves.

Zinc, Oxide, Evaporation, Oxidation

Resumen

El siguiente proyecto se muestran los resultados de experimentos relacionados con la obtención de nanopartículas de zinc (Zn), sobre substratos de vidrio o alúmina por el proceso de evaporación térmica en un horno tipo tubular de alta temperatura a 800°C, con un flujo de 400 sccm de gas inerte (Argón) utilizado como gas de arrastre, para depositar las partículas de Zn metálico en la zona fría del horno, así mismo se trató térmicamente las muestras de depósito de Zn metálico con la intención de ser utilizadas como una propuesta material semiconductor tipo n basado en óxidos metálicos, el cual fue formado a través de la técnica de Oxidación térmica en el mismo horno tubular de lata temperatura a 500°C, con flujo de aire a 600 sccm para formar un ambiente oxidante y en consecuencia formar capas continuas de material de Óxido de Zinc (ZnO) y ser analizadas morfológicamente con Microscopia electrónica de Barrido, estructuralmente con Difracción de rayos X y eléctricamente colocando contactos metálicos a las muestras para medir sus curvas de Corriente Voltaje.

Zinc, Oxido, Evaporación, Oxidación

1. Introduction

At present we live surrounded by diverse equipment and technological devices, based on different materials, one of the most outstanding being semiconductors, which offer us great advances in the constant development of electronics, leading to the miniaturization of faster and faster devices. and better, without a doubt impacting our quality of life, conventionally the materials used for the design of these devices are silicon and germanium.

A semiconductor is any material that can act as a conductor or insulator depending on the input excitation, such as temperature, luminescence, atomic structure or the electric field, allowing different operating states to be presented, such as the conduction state when switching is allowed. passage of an electrical current or as an insulator preventing the flow of electrical current, used for embedded device applications (Choudhury, 2020).

The process of obtaining these semiconductor materials is conventionally developed with techniques such as the Czochralski Method, photolithography, ion implantation or cathodic sputtering, which involve robust equipment and are carried out in specialized facilities. (Ali, 2020).

This paper deals with the use of metallic oxide materials which can emulate the behavior of conventional semiconductor materials, thanks to their structural defects and can be used as a contribution for future projects in the development of integrated devices with different types of materials. that can be generated in the laboratory with the techniques of evaporation and thermal oxidation (Mikhlif, 2021).

It was chosen to work with metallic oxides due to their easy obtaining and especially zinc oxide (ZnO) due to its N-type electrical characteristics and its wide availability, in order to contribute to the development of materials technology and the introduction of options for work different materials in applications such as semiconductor to nanotechnology, where the synthesis and characterization of micro and nano structures of zinc oxide (ZnO) (Alsultany F. H., 2016).

1.2 Zinc oxide

Metal oxides with structures in the nanometer range have been widely investigated for their potential applications in sectors such as electronics, optics, materials science and the biomedical sector. Among these, zinc oxide (ZnO) is a key material in the industry due to its versatility, high chemical stability, its high electron transport capacity and its optical, electronic, magnetic and mechanical properties derived from nanometric spatial confinement (Dev, 2021). It is a biocompatible and relatively cheap material and can be functionalized, which allows it to expand its potential medical applications. The most common crystalline phase of this material is hexagonal wurtzite and it has a band-gap of 3.37 eV, which makes it suitable for short-wavelength optoelectronic applications. The lack of center of symmetry in this phase of wurtzite (Bakhsheshi-Rad, 2017), in combination with its high electromechanical coupling, it results in strong piezoelectric and pyroelectric properties and consequently its use in a wide range of applications. Much of the applied research has been on dye-sensitized solar cells, piezoelectric nanogenerators, sensors, optoelectronic and emission devices, photocatalysts, actuators in biomedical sciences, energy, and spintronics. Current knowledge of the physical and chemical properties of zinc oxide has not revealed that doping ZnO with rare earths generates new ferromagnetic properties. (Bagga, 2018).

1.3 Thermal Evaporation

Thermal evaporation is a physical vapor deposition (PVD) process, usually in high vacuum (10-5 torr or higher vacuum values), (Hamelmann, 2016), in which atoms or molecules reach a substrate from a vaporizing heat source without collisions in the deposition chamber. This process is one of the simplest and oldest when it comes to thin film deposition, (Kamalianfar, 2021). The material is placed on a source which is heated by some thermal or electrical process, such as a resistor, high-temperature oven, eddy current, electron beam, laser beam, or arc discharge. The evaporated particles are transported and are deposited on a substrate that normally serves as a support to control the evaporated films (Mora Viquez, 2022). The vapor expands inside the chamber that contains the substrate, and subsequently condenses on the substrate that is kept at a lower temperature (Kumar R. K.-D., 2015). Vacuum evaporation involves thermodynamic considerations, such as phase transitions from which the equilibrium vapor pressure of materials is derived, and kinetic aspects of nucleation and growth. The latter is important in the evolution of the microstructure of the deposited layer. The understanding of the theory of the evaporation process is based on the kinetic theory of gases (Liu, 2018).

1.4 Materials and methods Thermal Evaporation

As the first part of this evaporation process, a commercial bar of zinc (Zn) with a purity of 99.999% was used, from which the target was obtained for thermal evaporation, roughing the bar in a controlled manner to obtain the zinc filings (Zn), for the zinc (Zn) evaporation process, a horizontal tube resistive furnace (CMOD-HAT-1100D25) was also used. To obtain and control the layers of evaporated metallic zinc (Zn), a support such as glass or alumina substrates was used, which were conditioned to adjust to the conditions of the quartz tube.

A quartz tube for the horizontal furnace whose length is 60 cm with a radius of 1 cm and a thickness of 1 mm was used as a reaction chamber for oxidation and thermal evaporation. In addition, 2 lids were printed with 3D printing. the flow of inert gases or air inside the reaction chamber for which they were designed with the help of SolidWorks software and later printed on a 3D printer (Anet A8 open source Prusa i3 3D printer).

The deposition of thin films by thermal evaporation is a technique that complements the thermal oxidation technique to obtain oxides. Zinc (Zn) filings with a purity of 99.999% were used in this deposition process. Zn is also an element of the transition metal family and has a compact Hexagonal structure and a melting point equal to 419.5°C. In this case, the deposition is carried out at a temperature of 800°C with a flow of Industrial Argon gas. This gas, being inert, was used to transport the evaporated white Zinc (Zn) and take it to a lower temperature zone within the reaction chamber, likewise it prevents oxidation due to the effect of temperature.

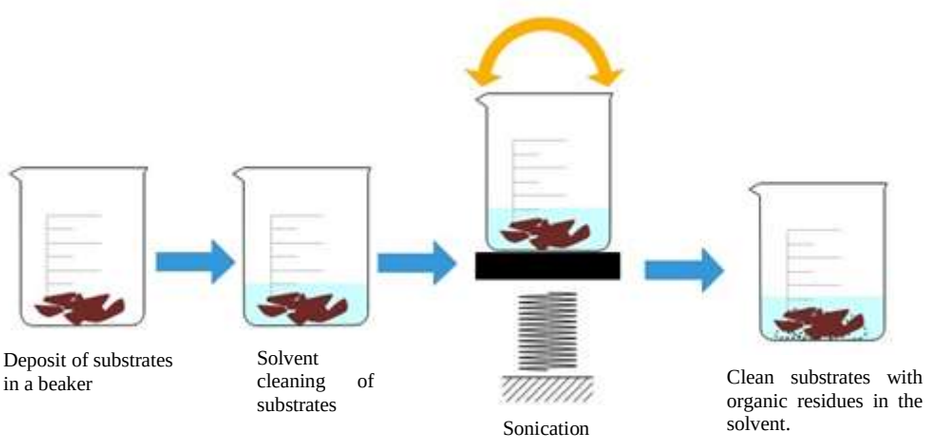
1.4.1 Substrate preparation, washing and degreasing

For this process, a sonicator equipment was used to facilitate the washing of the supports used, with the aim of eliminating organic and inorganic residues that it may have and with this, have surfaces free of parasitic elements. For this, the substrates were washed with three different solvents, which are:

1. Xylene C6H4(CH3)2: colorless and flammable liquid chemical compound, used in the process to remove organic residues from the samples.
2. Acetone (CH3)2CO: liquid, colorless, flammable and volatile organic compound, used to remove inorganic residues from substrates.
3. Methanol CH3OH: colorless liquid used to remove xylene and acetone residues in the samples.

The substrates are deposited inside a 50ml glass beaker, in which 20ml of each solvent mentioned above are added, to subsequently sonicate the substrates in the solutions for 15 minutes at a power of 50 watts. At the end of the time, each of the residues of the different solvents were discarded. This process is repeated with each of the solvents; xylene, acetone and methanol respecting the order respectively shown in figure 1.1

Figure 1.1 Substrate preparation



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1.4.2 Oven preheat

In this step it is necessary to preheat to a temperature of 800°C and wait until the temperature with the PID controller of the horizontal tube resistive furnace (CMOD-HAT-1100D25) is stable in order to start the thermal evaporation process, in the Figure 1.2 shows the horizontal tube furnace, which has a length of 40 cm, a width of 20 cm and a height of 20 cm.

Figure 1.2 Horizontal Tube Resistive Furnace (CMOD-HAT-1100D25)

Source of Consultation: Prepared by the work team

1.4.3 Preparation of the White of zinc filings (Zn)

In order to carry out the thermal evaporation of Zinc (Zn) it is necessary to obtain the target for evaporation, through a commercial zinc bar filing, in this process 120 mg of zinc (Zn) filings were used, which was deposited on a quartz canoe as shown in figure 1.3 to contain the evaporation target and withstand the evaporation temperature.

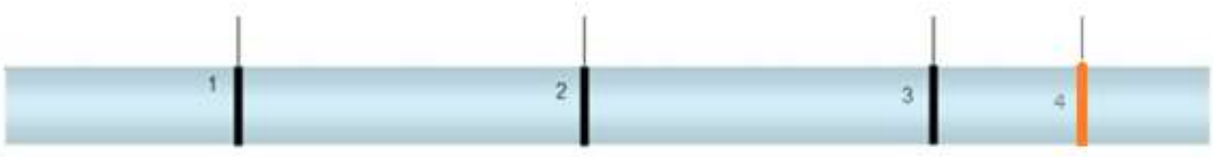
Figure 1.3 Zinc (Zn) filings on the quartz canoe

Source of Consultation: Prepared by the work team

1.4.4 Mounting of the zinc blank and substrate on the quartz tuve

In this phase, the quartz tube was used that will serve as the reaction chamber, whose dimensions are 60 cm long with a radius of 1 cm and a thickness of 1 mm between the walls, in this tube the zinc filings are introduced (Zn) that was previously deposited on a quartz canoe, until the zone of highest temperature of the tubular furnace to be evaporated, this zinc (Zn) filings must remain in the middle of the quartz tube with the help of a stainless steel rod. In figure 1.4, marks 1 and 3 are shown, which indicate the limit of the walls of the horizontal tube resistive furnace, so the canoe containing the zinc (Zn) filings will be located at mark number 2, while at mark number 4 which is 25 cm from the center of the oven and is where the previously washed and degreased substrate will be placed.

Figure 1.4 Quartz tube with markings for mounting the zinc filings and the substrate



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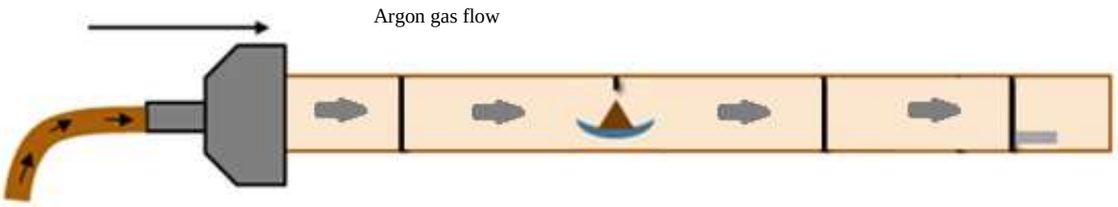
1.4.5 Coupling of the lid with the quartz tube

One of the printed covers is placed on the quartz tube at the left end, which has a hole with seals in the center that allows the access of gases to the tube as a chamber and a hose for the supply of carrier gas is placed. In this phase, a hose is connected to the lid coupled to the quartz tube so that it is supplied with argon gas, which is regulated with a flowmeter at 400 sccm as shown in figure 1.5.

Figure 1.5 Carrier gas supply (Argon)



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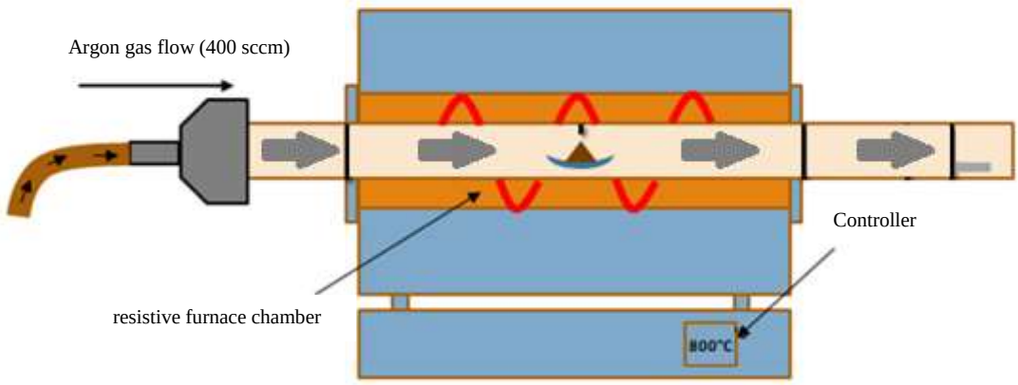
1.4.6 Thermal Evaporation Phase

In this phase, the quartz tube is placed inside the high-temperature resistive furnace with a horizontal tube previously heated to 800°C, and the canoe containing the zinc (Zn) shavings must already be placed inside the quartz tube, as well as the substrate on which the evaporated zinc (Zn) will be deposited, as shown in figure 1.6. Before the quartz tube is placed inside the furnace, the carrier gas (Argon) must already be flowing. Once the quartz tube is placed, the zinc (Zn) thermal evaporation process lasts 30 min with argon gas flow at 400sccm.

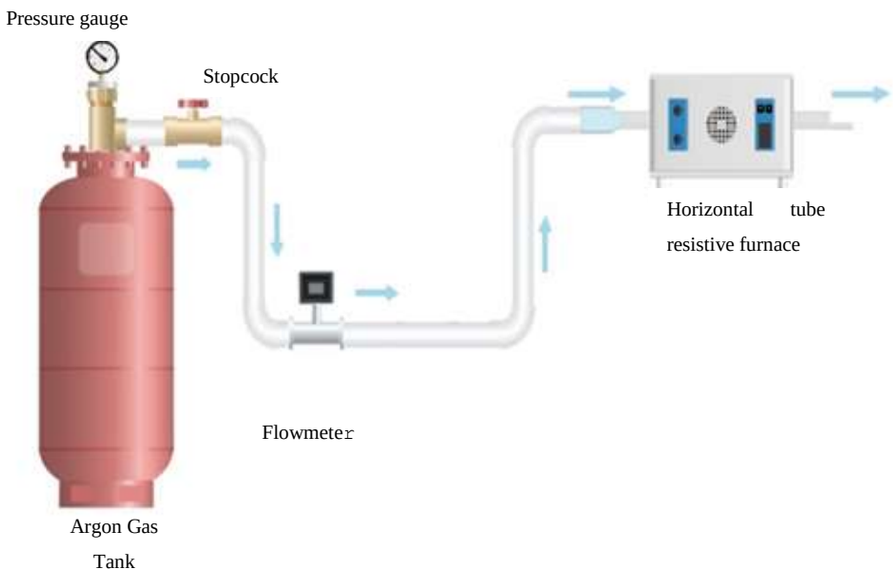
Figure 1.6 Thermal evaporation of zinc (Zn)



Source of Consultation: Prepared by the work team

Figure 1.7 schematically shows the interconnected equipment to carry out the thermal evaporation process of zinc, which must be calibrated and monitored by users trained in handling them to avoid wasting supplies or changes in the obtaining process.

Figure 1.7 Thermal evaporation system for zinc (Zn).



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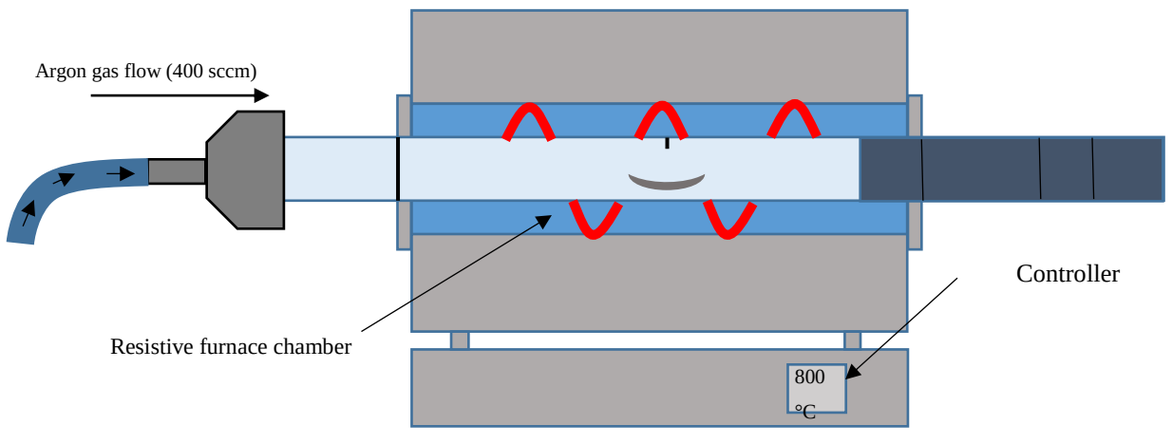
1.4.7 Disassembly of the quartz tube

Evaporation inside the quartz tube occurs as the blank is consumed in the flat area of the furnace and the evaporated particles are transported by the carrier gas to a lower temperature area, depositing the material in a metallic form on the walls of the furnace. the camera and at the same time on the previously placed substrate as shown in Figure 1.8. Once the thermal evaporation time is over, the cooling is abrupt, that is, the quartz tube is removed from the horizontal tube resistive furnace, once the quartz tube is out, it is necessary to close the flow of the carrier gas (Argon) and subsequently disassemble the substrate on which the evaporated zinc (Zn) was deposited.

Figure 1.8 Dismantling of the zinc (Zn) thermal evaporation system



Source of Consultation: Prepared by the work teamtrabajo

1.5 Thermal oxidation

Currently there are several proposals on the growth mechanism of the various structures for zinc oxide (ZnO). The vapor-solid mechanism is the most used to understand the formation of these, although the vapor-liquid-solid mechanism is also taken as a basis depending on the temperatures used in the formation of metal oxide. (Kumar R. K.-D., 2015). Reported temperatures for nanostructure formation in the literature range from 400 degrees to 1000 degrees Celsius based on the fact that zinc has a melting point of 420 degrees Celsius and a boiling point of 907 degrees Celsius. Depending on the temperature range used, it is the growth mechanism that tends to be used to explain the formation of the structures. The decomposition of ZnO happens at 1400 degrees, so it does not tend to exceed or reach these temperatures (Płóciennik, 2015).

1.6.1 Materials and methods Thermal Oxidation

For the thermal oxidation of metallic zinc, the same horizontal tube furnace was used to give a thermal treatment to the samples obtained in the evaporation process at 500 ° C for two hours and in the same way the quartz tube was used as a reaction chamber. to produce thermal oxidation and an air compressor was added to the production system to make an air flow flow inside the chamber and thus create an oxidizing environment for obtaining zinc oxide (ZnO).

1.6.1 Oven preheat

For this phase of thermal oxidation it is essential to preheat the horizontal tube resistive furnace to 500°C and wait for the PID controller of the furnace to reach the temperature and be stable to start the thermal oxidation process.

1.6.2 Mounting of the substrate in the quartz tube

In this phase of the process it is necessary to use a quartz tube similar to the one used in thermal evaporation, the tube has a length of 60 cm, a radius of 1 cm and a thickness of 1 mm between its walls, within In this quartz tube, the evaporated zinc sample was placed on the substrate, right in the middle of the tube. A lid was placed on the quartz tube at the left end, which was connected to a hose to supply air and achieve an oxidizing environment inside the reaction chamber and efficient thermal oxidation process, as shown in the figure. 1.9.

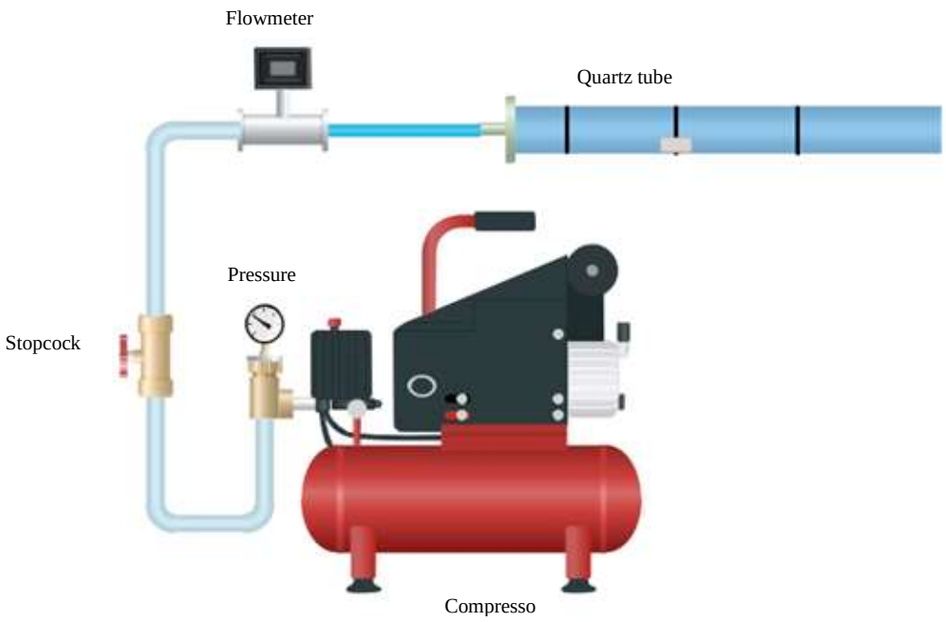
Figure 1.9 Mounting of the substrate in the quartz tube

Source of Consultation: Prepared by the work team

1.5.4 Air Supply Coupling

Figure 1.10 schematically shows the interconnected equipment, to carry out the thermal oxidation process of zinc, where a commercial air compressor was incorporated, to maintain a constant flow and favor oxidation inside the chamber, all This equipment must be calibrated and monitored by users trained in handling it to avoid wasting supplies or changes in the procurement process.

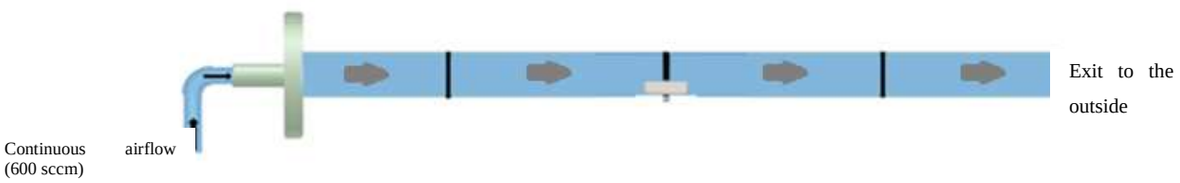
Figure 1.10 Thermal oxidation system of zinc (Zn)



Source of Consultation: Prepared by the work team

What shown in Figure 1.11, a hose is connected to the lid coupled to the quartz tube to be supplied with air, which is regulated with a flowmeter at 600 sccm.

Figure 1.11 Air supply



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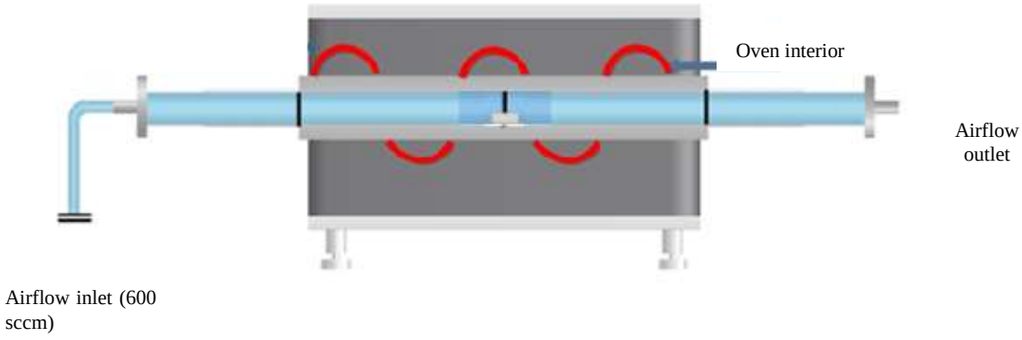
1.5.5 Thermal Oxidation Phase

In this phase, the quartz tube is placed inside the horizontal tube resistive furnace previously heated to 500°C, inside the quartz tube the sample obtained in the process of thermal evaporation of zinc (Zn) must already be placed, as shown. in Figure 1.12. Before the quartz tube is placed inside the oven, the air supply must already be flowing. Once the quartz tube is placed, the zinc (Zn) thermal oxidation process lasts 2 hours with air flow at 600 sccm. Once the thermal oxidation time is over, the air flow is closed and cooling is natural, that is, the quartz tube is removed from the horizontal tube resistive oven until the oven is at room temperature again once the cooling tube is out. quartz will disassemble the substrate, obtaining as a result the sample of zinc oxide (ZnO).

Figure 1.12 Thermal oxidation process



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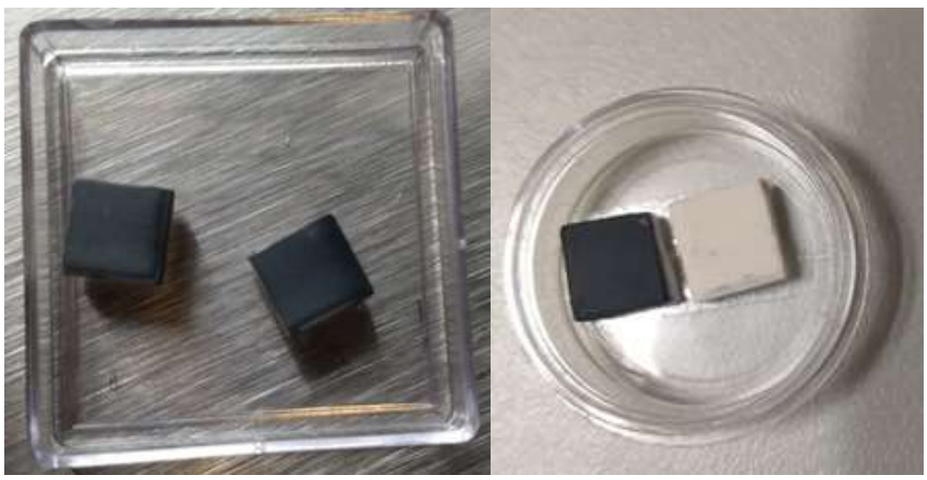
1.6 Results

This section explains the results of the samples obtained with the previously described methodologies of evaporation and thermal oxidation, they were used to validate the technical results of characterization such as Scanning Electron Microscopy (SEM or SEM), X-Ray Diffraction Spectroscopy (XRD), characterization I-V, used to characterize oxides as semiconductors.

Figure 1.13 shows the physical results of the application of the methods used to obtain the Zinc Oxide material, where on the left side of the Figure the result of the deposition of the thermal evaporation of metallic Zinc on the substrates is observed. They serve as supports for the formation of the thin layer. On the right side of the Figure, the comparison and application of the second technique used, thermal oxidation, where the sample is observed before the oxidation process exhibits a dark grayish coloration and after thermal oxidation its coloration of the sample changes to whitish, typically as zinc oxide (ZnO) with large amounts of mass is known.

Figure 1.13 Results of the evaporation and thermal oxidation process.



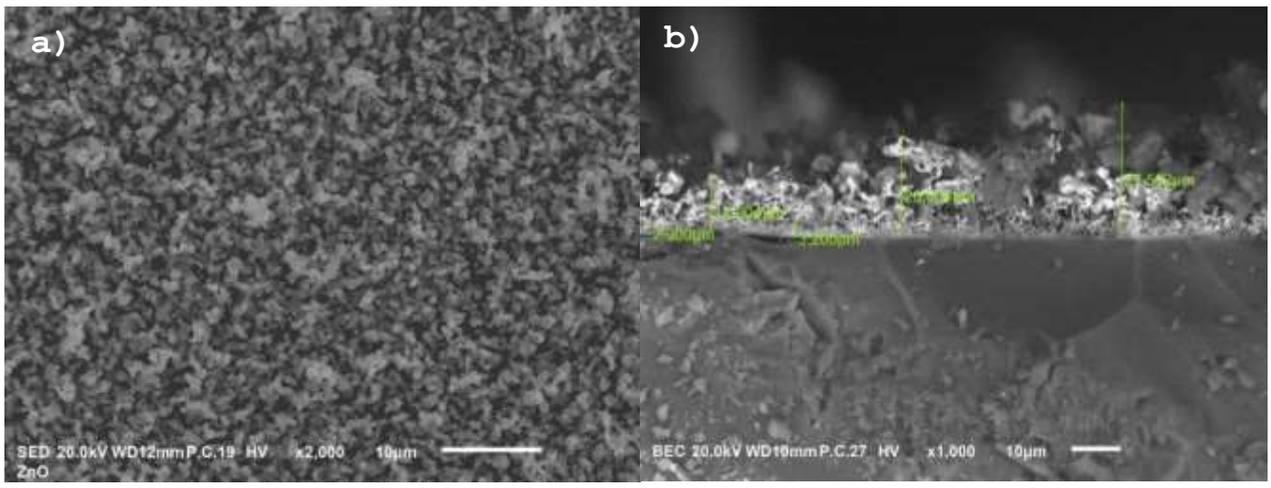
Source of Consultation: Prepared by the work team

1.6.1 Morphological characterization

The first scanning electron microscopy technique SEM for its acronym in English Scanning Electron Microscope, consists of making an electron beam fall on the sample that is analyzed to obtain a detailed image of the sample surface. In this way we can see if the surface is continuous, compact, rough, smooth or porous. Scanning electron microscopy images were obtained with the help of the SEM scanning electron microscope equipment (SEM, JEOL JSM-6510LV).

Figure 1.14 shows a surface and lateral image of the substrate with a thermally treated zinc oxide deposit, the measurement was made at a distance of 10 µm and it is operated with a 20 kV electron beam, with a working distance (WD) 12 mm, in the same way the way in which the ZnO film was formed is observed, which is continuous and grows on the substrate (a). In image (b) a micrograph of one side of the sample was taken to measure the thickness of the film, this micrograph allows us to see that the film has a base layer of 3µm and from this the structure grows about 30µm in the thickest edges and 12µm at the thinnest edges, you can even notice that hexagonal structures begin to form at some points in the film.

Figure 1.14 Morphology (a) and thickness (b) of the film that forms



Source of Consultation: Prepared by the work team

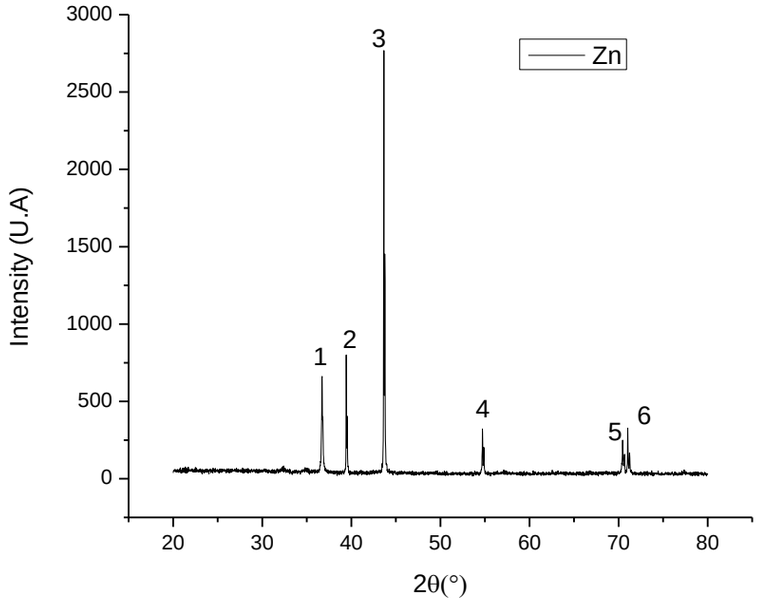
1.6.2 Structural characterization

X-Ray Diffraction (XRD) characterization consists of an X-ray beam that penetrates the surface of a sample and is then diffracted by its crystal planes. The characteristic diffraction angle and intensity of a crystalline structure is unique for each material, so X-ray diffraction in polycrystalline samples allows identifying qualitative and quantitative aspects to determine physical and chemical properties (Van Khai, 2018).

The samples obtained were analyzed before and after the thermal treatment by X-ray diffraction (XRD) with the help of the X-ray diffractometer measurement equipment (XRD, Bruker D8 discover), under the conditions; CuKα radiation (1.5418 Å), 40 KV and 40 mA in a 2θ range from 20° to 80° with an increment of 0.0200° and an incidence time of 8s.

Graph 1.1 shows the result before the thermal oxidation process, showing the corresponding peaks of 1-6 at 2θ= 36.7199°, 39.4400°, 43.6601°, 54.7200°, 54.8801°, 70.4599°, 70.6800°, 71.0400° and 71.2402°, which correspond to metallic zinc obtained thanks to thermal evaporation.

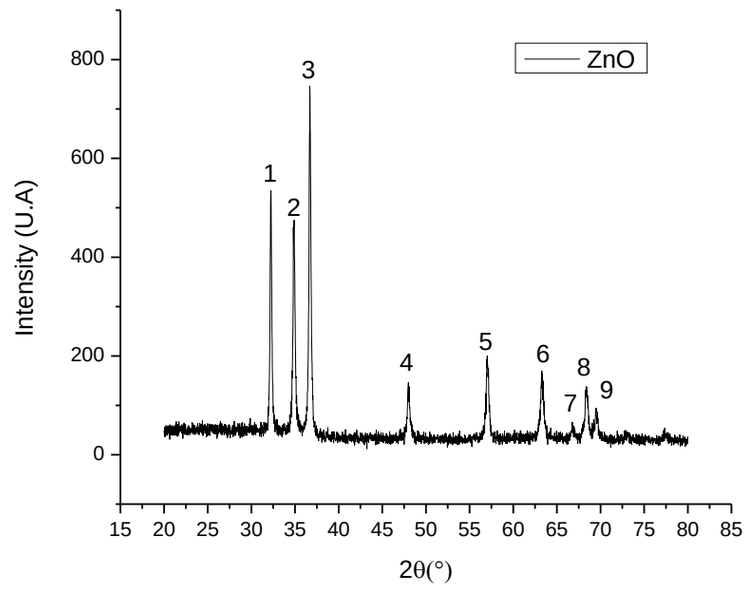
Graphic 1.1 Diffractogram of evaporated zinc (Zn)



Source of Consultation: Prepared by the work team

Graph 2 shows the results of the structural characterization once a sample is exposed to the thermal treatment to thermally oxidize, the result after the thermal oxidation process can be seen reflecting in the corresponding diffraction peaks of 1-9 at 2θ= 32.2464° , 34.8206°, 34.8999°, 36.700°, 47.9793°, 48.0802°, 56.9834°, 63.2693°, 66.7539°, 68.3710°, 69.4300|, 69.6579°, 72.8738°, 77.3105°, these values were compared with the crystallographic table ZnO from PDF 00-036-1451, therefore, it is confirmed that the sample is only ZnO, where the orientations of the peaks were shown and are shown below peak 1: (1 0 0), peak 2: (0 0 2), Peak 3: (1 0 1), Peak 4: (1 0 2), Peak 5: (1 1 0), Peak 6: (1 0 3), Peak 7: (2 0 0), Peak 8: (2 0 1), peak 9: (2 0 1)

Graphic 1.2 Diffractogram of zinc oxide (Zno)



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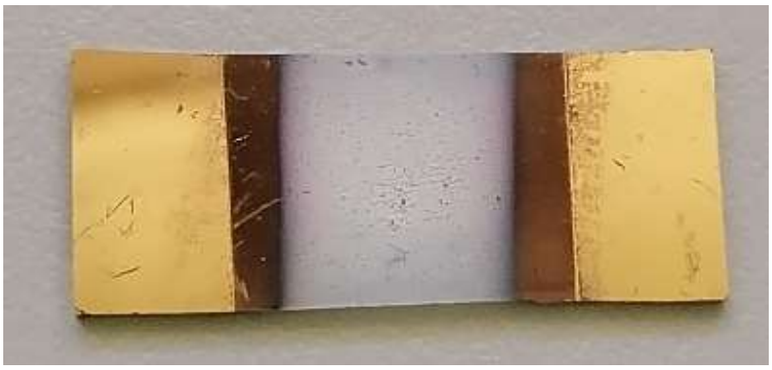
1.6.3 Electrical Characterization I-V

The electrical characterization current and voltage I-V, is used to know the operation of the constructed ZnO device. However, in order to carry out the characterization it is necessary to add electrical contacts to the structure. These contacts must be ohmic, that is, they must offer a low resistance, and must be capable of conducting a current flow in both directions in order to be able to bias both forward and reverse (Van Khai, 2018). I-V characterization also serves to identify whether the junction that forms between the metal and the semiconductor forms an ohmic or rectifying contact. For our zinc oxide (ZnO) samples, it was necessary to add gold (Au) contacts by sputtering with the help of the Agar Auto Sputter Coater (Model.108A) equipment, starting from a 99.995% pure Gold target, a diameter of 57mm and thickness of 0.1mm, with 30 mA of current to generate the arc and a distance of 5 mm from the target to the base, leaving a distance of 1 cm between the gold (Au) contacts that were placed on the gold oxide. zinc (ZnO), as shown in Figure 1.15.

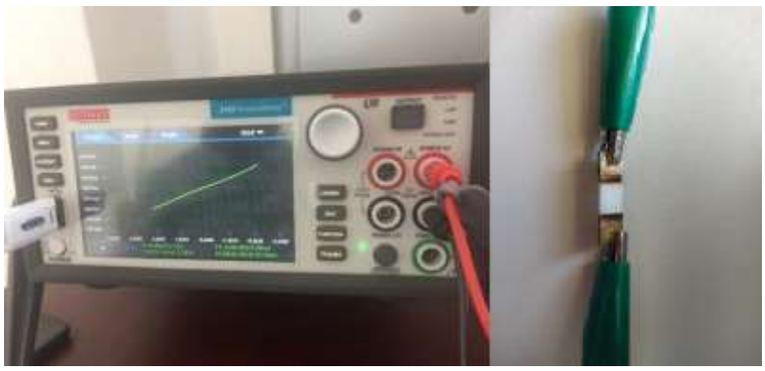
Figure 1.15 Sample of zinc oxide (ZnO) with gold (Au) contacts.



Source of Consultation: Prepared by the work team

Figure 1.16 shows the electrical measurements made to the Au/Zinc Oxide/Au sample, which were made with the help of a KEITHLEY 2450 source with which a sweep was made in a range of -20 volts to 20 volts. , with a step of 10 mV and a current limit of 500 mA, in order to obtain the characteristic curve of the zinc oxide (ZnO) sample.

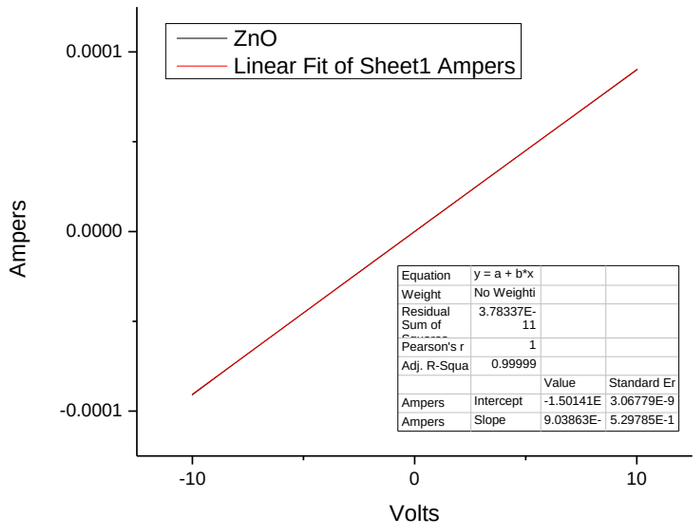
Figure 1.16 I-V measurement



Source of Consultation: Prepared by the work team

As shown in the IV curve of graph 1.3, it reflects an ohmic behavior of the sample, which exhibits a linear proportional form in both polarities and has a resistance R=1.10636 MΩ per centimeter of sample, corresponding to zinc oxide. measured.

Graphic 1.3 Characteristic curve of this sample of zinc oxide (ZnO)



Source of Consultation: Prepared by the work team

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1.8 Conclusions

Using the two methodologies, it was possible to obtain controlled layers of Zinc Oxide where the process could be validated, characterizing the samples morphologically, structurally and electrically.

As a relevant fact to obtain metallic Zn films, it is necessary that the deposition is carried out in an inert environment where the carrier gas does not react with the target, in this way the films obtained are continuous and uniform, but porous. The thickness of the zinc (Zn) film is dependent on the gas flow, the distance between the target and the substrate, in addition this same thickness is related to the deposition time to which the sample is exposed, as well as dependent of the amount of Zinc that evaporates in the blank and of the evaporation temperature.

The choice of the electrical contacts that were coupled to the device to characterize it electrically must be ohmic so that the current flows without encountering resistance between the metal and the semiconductor, thus not affecting the I-V measurement.

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