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INVESTIGATION OF THE EFFECT OF DOPED NICKEL ON THE STRUCTURAL SURFACE AND OPTICAL PROPERTIES OF ZnO THIN FILMS PREPARED BY THE SILAR METHOD

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AMMAR HAMDAN DHEYAB DHEYAB

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By Ammar Hamdan Dheyab DHEYAB

January 2024

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ABSTRACT

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Ammar Hamdan Dheyab DHEYAB

Master of Science in Physics

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January 2024

Thin film technologies play a significant role in the development of modern electronic and optoelectronic devices. These technologies attract considerable interest in many application areas by enabling the production of high-performance and low-cost materials. Zinc oxide (ZnO) thin films stand out as a potential material for various electronic and optical devices. Thin film technologies in this field promise high efficiency and low-cost devices. ZnO is a semiconductor material with a wide energy range, making it an attractive option for use in electronic and optoelectronic devices due to its optical properties and high electron transport capabilities. Additionally, the physical and chemical properties of ZnO indicate a wide range of potential applications, from surface coatings to sensors in various industrial applications. Especially, doped ZnO semiconductor materials grown on FTO (fluorine-tin-oxide) substrates are significant candidates for many applications such as solar cells, gas sensors, and biosensors.

In this thesis study, undoped FTO/ZnO and Nickel (Ni) doped, Ni 1% FTO/ZnO, Ni 2% FTO/ZnO, and Ni 3% FTO/ZnO thin films prepared using the SILAR (Successive Ionic Layer Adsorption and Reaction) method were investigated. The influence of nickel's contribution on the structural, surface, and optical properties of the produced films was particularly studied. Structural analyses were conducted using techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM). The findings indicate significant changes in the crystal structures and surface morphologies of nickel-doped FTO/ZnO thin films. Additionally, the optical properties were examined using UV-Vis spectroscopy, and the impact of nickel on the film's optical characteristics was thoroughly evaluated. Nickel doping levels in the ZnO thin films were altered from 1% to 3%. SEM-EDS analyses revealed that the produced ZnO thin films formed cluster-like structures. It was observed that the structures varied depending on the nickel content. Surface homogeneity distribution was most evident in the FTO/ZnO-Ni 3% sample among the produced specimens. XRD analyses identified the dominant diffraction peaks of the samples as SnO_2 (FTO-substrate). Overall, the samples exhibited a tetragonal crystal structure with orientations of (101), (202), and (211). As the Ni-doped concentration increased, an increase in peak intensities and a decrease in half-peak widths in the films were observed. These results demonstrate the potential use of the produced samples in solar cell technologies, semiconductor-based gas sensor fabrication, and similar fields.

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Keywords: SILAR method, Zinc oxide, Thin films, FTO, Ni-doped

ÖZET

SILAR METODU İLE HAZIRLANAN FTO/ZNO İNCE FİLMLERİNİN YAPISAL YÜZEYSEL VE OPTİK ÖZELLİKLERİ ÜZERİNDE NİKEL KATKISININ ETKİSİNİN İNCELENMESİ

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İnce film teknolojileri, modern elektronik ve optoelektronik cihazların geliştirilmesinde önemli bir role sahiptir. Bu teknolojiler, yüksek performanslı ve düşük maliyetli malzemelerin üretimini sağlayarak birçok uygulama alanında büyük ilgi görmektedir. Çinko oksit (ZnO) ince filmleri, çeşitli elektronik ve optik cihazlarda kullanılmak üzere potansiyel bir malzeme olarak öne çıkmaktadır. Bu alanda, ince film teknolojileri, yüksek verimlilik ve düşük maliyetli cihazlar için umut vaat etmektedir. ZnO, geniş bir enerji aralığına sahip yarı iletken bir malzeme olup, optik özellikleri ve yüksek elektron taşıma özellikleri nedeniyle elektronik ve optoelektronik cihazlarda kullanımı için cazip bir seçenek sunmaktadır. Ayrıca, ZnO'nun fiziksel ve kimyasal özellikleri, farklı endüstriyel uygulamalarda yüzey kaplamalarından sensörlere kadar geniş bir yelpazede kullanım potansiyeline işaret etmektedir. Özellikle FTO (florine-tin-oksit) alltaş üzerine büyütülen katkılı ZnO yarı iletken malzemeler güneş hücreleri, gaz sensörleri ve biosensörler gibi birçok uygulamalar için önemli adaylardandır.

Bu tez çalışmada, SILAR (Succesive Ionic Layer Adsorption and Reaction) yöntemi kullanılarak hazırlanan katkısız FTO/ZnO ve Nikel (Ni) katkılı, Ni 1 % FTO/ZnO, Ni 2 % FTO/ZnO ve Ni 3 % FTO/ZnO ince filmleri incelenmiştir. Üretilen filmlerin özellikle yapısal, yüzeysel ve optik özellikleri üzerinde nikelin katkısının etkisi araştırılmıştır. Yapısal analizler, X-ışını kırınımı (XRD) ve taramalı elektron mikroskobu (SEM) gibi tekniklerle gerçekleştirilmiştir. Bulgular, nikelin FTO/ZnO ince filmlerinin kristal yapılarında ve yüzey morfolojisinde belirgin değişikliklere neden olduğunu göstermektedir. Ayrıca, optik özellikler UV-Vis spektroskopisi kullanılarak incelenmiştir ve nikelin filmin optik özelliklerini nasıl etkilediği detaylı olarak değerlendirilmiştir. Katkılı ZnO ince filmlerde Nikel (Ni) katkılama oranı % 1'den % 3'e değiştirilerek elde edilmiştir. SEM-EDS analizlerinden üretilen ZnO ince filmlerin kümeler şeklinde yapıların oluşturmuştur. Oluşan yapılar Nikel katkısına bağlı olarak değiştiği gözlendi. Üretilen numunelerden yüzey homojenliği dağılımın en yüksek FTO/ZnO-Ni% 3 numunesinde görüldü. Üretilen numunelerin XRD analizlerinden baskın kırınım zirveleri SnO_2(FTO-alltaş) elde edilmiştir. Numuneler genel olarak tetragonal kristal yapısını gösteren (101), (202) ve (211) yöneliminde olduğu görüldü. Ni-doped katkı miktarı arttıkça filmlerin pik şiddetlerinde artış ve yarıpik genişliklerinde azalma olduğu görülmüştür. Bu sonuçlar, üretilen numunelerin güneş hücresi teknolojileri, yarıiletken tabanlı gaz sensörü yapımı gibi alanlarda kullanılabilme potansiyeline sahip olduğu değerlendirilmiştir.

2024, 41 sayfa

Anahtar Kelimeler: SILAR metodu, Çinko oksit, İnce film, FTO, Ni-katkılama

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CONTENTS

LIST OF SYMBOLS

A	Absorbance value
α	Absorption coefficient
Å	Angstrom value
I_A	Absorbed intensity
θ	Bragg angle
k_B	Boltzman constant
°C	Centigrade degree
Cs	Crystal size
N	Complex refractive index
δ	Dislocation density
E_g	Energy gap
eV	Electron volt
d_{XRD}	Experimental distance
k_o	Extinction coefficient
ν	Frequency value
β	Half peak width
$M_{(hkl)}$	Miller indices
M	Molar value
nm	Nanometer value
d_{hkl}	Interplaner distance
h	Planck's constant
E_{ph}	Phonon energy
n_o	Refractive index
R	Reflectance
I_R	Reflected intensity
d_{CARD}	Standard distance
I_o	Standard (Incidence) intensity
K	Shape factor
T	Transmittance value
I	Transmittance intensity
TCO	Transparent conductive oxides
λ	Wavelength

LIST OF ABBREVIATIONS

EDS	Energy dispersive X-ray spectroscopy
FESEM	Field emission scanning electron microscope
UV-VIS	Ultraviolet-visible
XRD	X-ray diffraction

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1. INTRODUCTION

1.1 Overview of Nanotechnology

Nanotechnology is an interdisciplinary field focused on the design, fabrication, and control of materials, devices, and systems at the nanometer scale. This discipline aims to explore the unique properties of materials by scaling them down to the size of atoms and molecules and to enhance their applications (Akhtar et al., 2008). This next-generation technology is a multidisciplinary field that emphasizes the control, design, and fabrication of materials, components, and devices at the nanometer scale. Research in this field aims to discover the special properties of nanomaterials and expand their applications (Chang *et al.* 2012).

Nanotechnology emerges from the convergence of various fields such as nanoscience, materials science, physics, chemistry, biology, and engineering. This field is utilized to explore unique properties arising from the scale, high surface-area ratios, and optical, electronic, and magnetic characteristics (Raidou *et al.* 2010). Nanotechnology has the potential to revolutionize many areas, including electronics with smaller and more efficient devices, medicine with more effective drug delivery systems, energy production with more efficient solar cells, and water purification with more effective filters.

Nanotechnology has played a significant role in the advancement of semiconductor devices, bringing revolutionary innovations to this field. While semiconductor technology forms the foundation of modern electronic devices, nanotechnology has endowed this area with unique characteristics and enhanced performance (Zhang *et al.* 2010). In this context, the contributions of nanotechnology to the evolution of semiconductor devices are of great importance. Structuring materials like semiconductors at the nano scale enables electronic devices to be smaller, faster, more efficient, and more powerful. For example, transistors produced with nanotechnology have enabled the development of higher-performance and energy-efficient microchips (Sutton *et al.* 2007).

These technological advancements have enabled modern electronic devices such as computers, cell phones, tablets, and other mobile devices to shrink in size while increasing their performance (Willander *et al.* 2009). Moreover, semiconductor devices produced through nanotechnology have revolutionized technological advancements by offering benefits such as faster data transfer, lower power consumption, and higher integration capacity (Zhongwei *et al.* 2021).

The development of semiconductor devices through nanotechnology also enables the emergence of new and innovative applications. Nanotechnology has expanded the boundaries of semiconductor devices and increased their potential in various fields such as biomedical devices, energy storage and conversion systems, sensors, and optoelectronic devices (Kim *et al.* 2004).

Zinc oxide (ZnO) thin films stand out as a significant nanomaterial with a wide range of applications, thanks to the opportunities provided by nanotechnology (Gao *et al.* 2004). ZnO owes its prominence in nanotechnology to various properties. Firstly, ZnO is a semiconductor material with a wide energy bandgap, offering a significant advantage in the development of various devices and sensors (Vu *et al.* 2019). Additionally, ZnO's high electron mobility and thermal stability make it effective in electronic applications such as light-emitting diodes (LEDs) and photovoltaic devices (Xiangdong *et al.* 2005). ZnO thin films have the potential to be a prominent material in nanotechnology. These films can be prepared through various methods for various applications in nanotechnology (Bougrine *et al.* 2003). Particularly, ZnO thin films created in atomic layers are of great importance for the formation of nanostructures and understanding their electronic, optical, and magnetic properties (Chen *et al.* 2000).

The extensive use of transparent and highly conductive thin films in both industrial and research applications has triggered research into semiconductor thin films (Bamiduro *et al.* 2014). As a result, there is growing interest in producing films containing ZnO and ZnO-based materials to reduce costs and introduce alternative materials. Research focusing on the structural properties of these produced films has garnered attention (Avcı *et al.* 2019).

Thin films can be produced using cost-effective methods. Due to their electrical and optical properties, the thin films produced are widely used in various applications such as solar cells, semiconductor photodetectors, and lasers (Mondal *et al.* 2013). Therefore, thin films are extensively researched in academic studies as well (Yoon *et al.* 2015). In the literature, studies focus on both doped and undoped thin films (Aksoy *et al.* 2019). Initially developed by Nicolau using the SILAR method, multilayered and single-layered films continue to be developed for use in various devices (Ahn *et al.* 2007).

Various methods have been employed to obtain ZnO thin films, including the SILAR method, sol-gel method, chemical electrochemical deposition method (CVD), radio frequency magnetron sputtering, thermal evaporation, ultrasonic spray (SP), and stepwise ionic layer adsorption reaction method (Chen *et al.* 2000).

In this study, the SILAR method was chosen for thin film production due to its ease of layer control, low cost, and ease of application. The structural, optical, and morphological characterization of the prepared films was conducted using UV-Vis, XRD, and SEM/EDS methods (Grandbois *et al.* 1999).

Examinations of ZnO thin films produced using the sol-gel method with different ratios of nickel (Ni) doping, via XRD analysis, revealed that the samples exhibited a polycrystalline structure with crystallization solely attributed to the ZnO structure (Polat 2009). It was observed that the surface roughness and grain sizes of the produced thin films increased depending on the doping ratio. Optical transmittance analysis of the thin films showed low transmittance in the UV region and high transmittance in the visible region.

In this thesis, undoped FTO/ZnO and FTO/ZnO Ni % 1, FTO/ZnO Ni % 2, and FTO/ZnO Ni % 3 doped films were fabricated using the SILAR method. The structural, morphological, and optical properties of the produced thin films were investigated using UV-Vis, XRD, SEM/EDS methods. The analysis revealed that the bandgap of the produced thin films ranged from 3.12 to 3.21 eV. The optical properties and morphologies were examined depending on the doping ratio.

The organization of this thesis is as follows: The first chapter presents an introduction to the study. The second chapter provides information on the band structure and material parameters of ZnO semiconductor. The third chapter reviews the electron conduction theory and conduction mechanisms of semiconductor materials. In the fourth chapter, details about the experimental setup and measurement and analysis techniques used are discussed. The fifth chapter discusses the experimental results obtained from UV-Vis, XRD, SEM/EDS measurements, along with their implications and outcomes. Lastly, the sixth chapter evaluates the findings of the conducted study.

2. GENERAL INFORMATION

2.1 Zinc Oxide (ZnO)

Semiconductor materials constitute a significant class of materials that form the foundation of modern electronic devices. Positioned between conductors and insulators in terms of electrical conductivity, semiconductor materials serve as the building blocks of various electronic devices, as well as finding applications in fields such as nanotechnology, photovoltaic systems, optoelectronics, and sensors.

The importance of semiconductor materials is particularly notable in the realm of electronic devices. These materials are utilized in the fabrication of numerous fundamental components, including transistors, diodes, integrated circuits, and solar cells. For instance, transistors serve as the basic building blocks for providing processing power to computers, and thanks to semiconductor materials, these devices have become smaller and more powerful. Another crucial aspect of semiconductors is the controllability of electric current. This feature enables the realization of functions such as signal processing, power amplification, and data storage in electronic devices. Additionally, doping semiconductor materials with dopants of varying properties allows for precise control over the material's conductivity and other electrical characteristics.

There are essentially two classes of semiconductors (Akhtar *et al.* 2008). These are: i) elemental semiconductors found in group IV of the periodic table, and ii) compound semiconductors formed by the combination of elements from groups III-V or II-VI. Elemental semiconductors consist of single atoms. Examples include silicon (Si) and germanium (Ge). For compound semiconductors with binary structures, examples such as ZnO and InAs are the most commonly used.

Elements in group IV, such as Si and Ge, form covalent bonds among themselves; each element has four valence electrons, and each atom forms covalent bonds with four neighbors. Valence electrons are the electrons located in the outermost orbit relative to the atomic nucleus and are called valence electrons. The electrical characteristics of substances can be defined by looking at the number of valence electrons. Materials with a small number of valence electrons, such as one, behave as conductive materials because they can easily give up electrons. Elements with three to five valence electrons behave as semiconductors. Elements with eight valence electrons are stable in structure and do not tend to give or take electrons, so they behave as insulators. Elements in group IV, such as Si and Ge, have similar characteristics. The resistivity of pure silicon is approximately 1.6 Ω/cm^3. Elements in group IV, such as Si and Ge, can be influenced by heat and light. This property has led to the establishment and development of the semiconductor concept when certain changes occur when an electric current is applied to these materials.

2.2 Defects in Semiconductors and Dopant Atoms in Semiconductors

Semiconductor materials form the foundation of electronic devices, and defects and dopant atoms in these materials are critical factors that determine the properties and performance of the material. Defects and dopant atoms in semiconductors have significant effects on the conductivity, optical properties, thermal conductivity, and other important characteristics of the material.

Defects in semiconductors can be defined as irregularities or deficiencies in the crystal structure. These defects can manifest as point defects (e.g., vacancies or excess atoms), linear defects (e.g., edges or dislocations), or planar defects (e.g., stacking faults or grain boundaries). These defects can have a significant impact on the electronic properties of the material and sometimes lead to undesirable electrical behavior.

On the other hand, dopant atoms in semiconductors result from the substitution or addition of a specific atom within the material structure. These dopant atoms can alter the conductivity properties of the material, leading to the formation of either n-type or p-type semiconductors. For instance, elements like phosphorus or arsenic provide excess electrons in n-type semiconductors, while elements like boron can create electron deficiencies in p-type semiconductors. Understanding defects and dopant atoms in semiconductors is crucial for manipulating and controlling the desired properties of the material. This understanding forms the basis for the design of electronic devices, solar cells, sensors, and many other application areas.

Before semiconductor materials can be used in electrical applications, it is necessary to increase their conductivity. Therefore, silicon material needs to be doped from external sources. Dopant materials, such as antimony, gallium, or arsenic, with a valence electron count of 5, are preferred depending on the application's requirements. These dopants are added in very small amounts.

By doping semiconductor materials, two types of materials are obtained: (i) n-type semiconductors and (ii) p-type semiconductors (Bougrine *et al*. 2003).

2.3 n-Type Semiconductor

Semiconductors, materials that lie between electrical conductivity and insulation, and n-type semiconductors play a significant role in this classification. N-type semiconductors are semiconductor materials with excess added or doped electrons. These types of semiconductors exhibit distinct properties in terms of electron transport capability and conductivity.

The fundamental characteristic of n-type semiconductors is the presence of excess electrons provided by dopant materials. Typically, fifth-group elements such as phosphorus (P), arsenic (As), or antimony (Sb), which carry an excess of electrons, are used to create n-type semiconductors (Figure 2.1). These dopant materials settle into the crystal structure, creating free electrons within the material, thereby increasing its electrical conductivity.

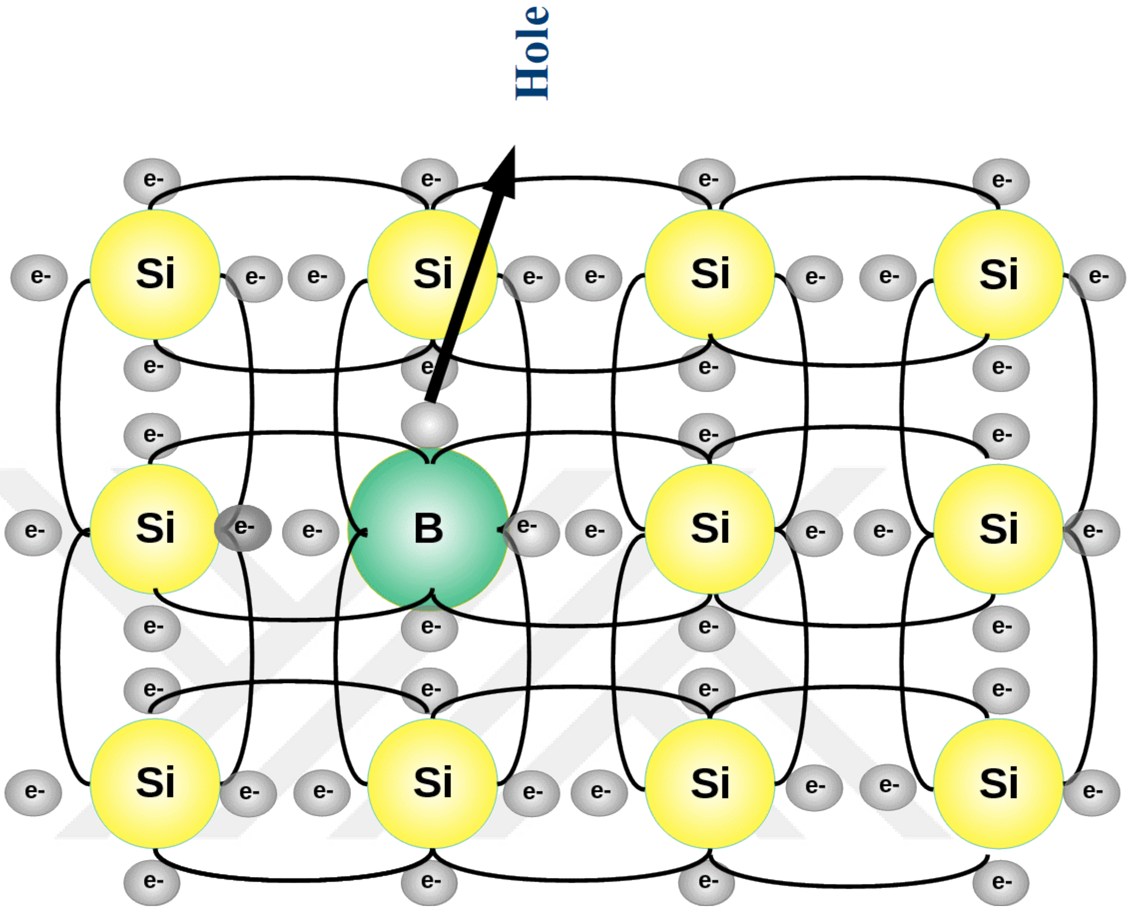

Figure 2.1 Formation of n-type semiconductor (Araki *et al.* 2004)

The presence of these excess electrons determines the properties of n-type semiconductors. The movement of electrons enables the flow of the material from one end to the other, allowing the formation of electric current. N-type semiconductors are particularly used in electronic devices and energy applications such as solar cells. For example, in transistors, n-type semiconductors function as regions where electrons can move easily, playing a significant role in circuit operation.

Understanding and using n-type semiconductors is important for the development and enhancement of electronic devices. By studying the properties of these materials, such as electrical conductivity, thermal properties, and other performance criteria, it is possible to develop new and improved technologies.

2.3 p-Type Semiconductor

Semiconductors lie between electrical conductors and insulators, with p-type semiconductors holding a significant place in this classification. P-type semiconductors are materials containing deficient electrons or "holes." Due to this electron deficiency, these semiconductors exhibit special properties in terms of electron transport capability and conductivity.

The fundamental characteristic of p-type semiconductors is the presence of deficient electrons or "holes" provided by dopant atoms. Typically, third-group elements such as boron (B), gallium (Ga), or aluminum (Al), which carry electron deficiencies, are used to create p-type semiconductors (Figure 2.2). These dopant atoms, when integrated into the crystal structure, create deficient electrons within the material, resulting in the formation of "holes," which are positively charged vacancies.

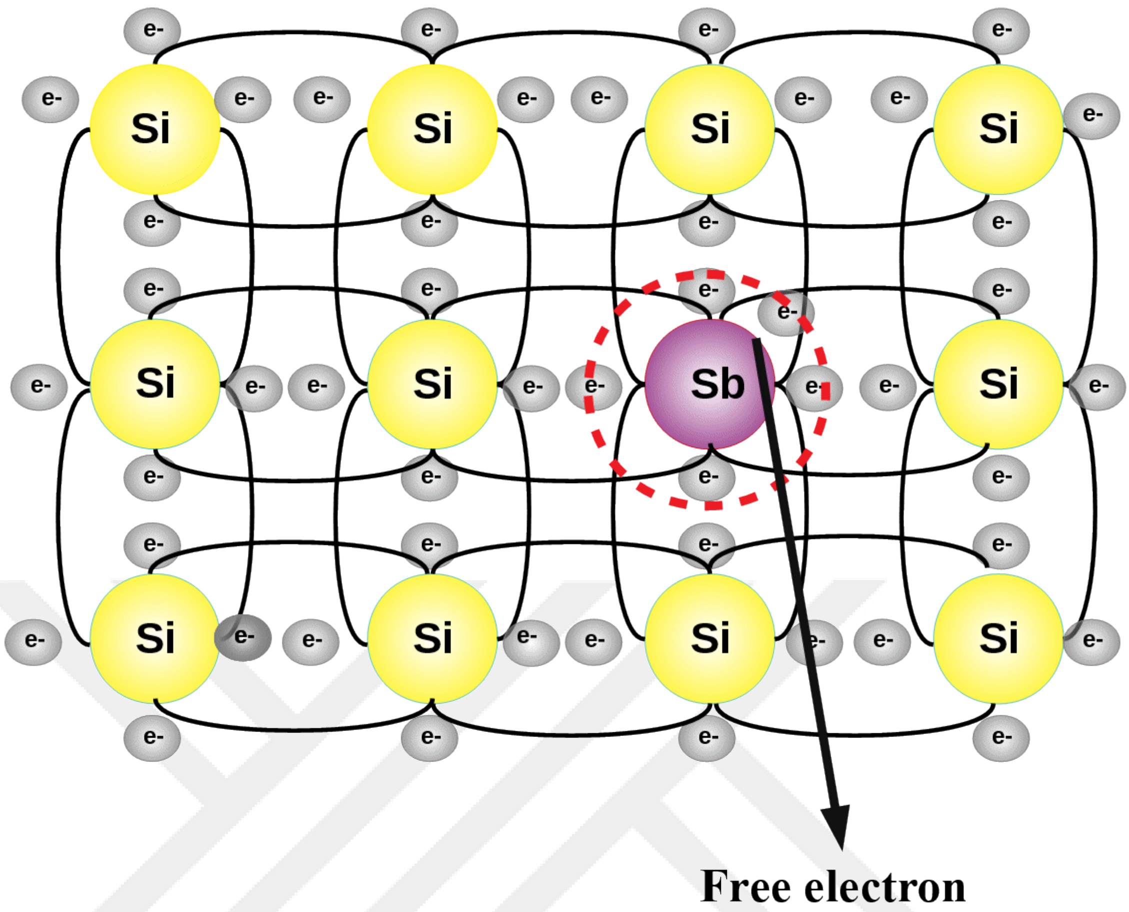

Figure 2.2 Formation of p-type semiconductor (Klein *et al.* 2020)

These holes or deficient electrons determine the properties of p-type semiconductors. The electron deficiency facilitates the movement of electrons within the material, allowing the formation of electric current. P-type semiconductors are particularly used in transistors and electronic devices requiring power amplification. These semiconductors play a significant role by facilitating easier electron movement in specific regions of circuits (Abdel-Wahab 2021).

The resulting new silicon structure, due to the deficiency of electrons, tends to accept electrons from the external environment. This type of structure is called an acceptor. The resulting structure is referred to as p-type semiconductor within this lattice.

Technological applications can be achieved using n-type and p-type semiconductor materials by employing the appropriate doping atoms. In n-type materials, excess electrons are freely available within the crystal structure, while in p-type materials, voids known as holes are formed within the crystal lattice. Thus, in n-type semiconductors, majority charge carriers are electrons, whereas in p-type semiconductors, majority charge carriers are holes. These majority charge carriers are the materials that enable controlled current flow through the semiconductor.

2.4. The Structure and Properties of ZnO Compound

ZnO compound is preferred in many technological applications due to its abundant presence in nature, high exciton binding energy, high ionicity, and a wide direct bandgap (Taner *et al.* 2011). The properties of ZnO are significant as a material because of its potential to control and adjust its optical, electrical, and chemical properties when applied as a thin film. This potential finds extensive use in various industrial and scientific fields, particularly in areas such as photovoltaic devices, semiconductor devices, sensors, imaging technologies, and nanotechnology.

2.4.1. Crystal structure

Most II-VI compound semiconductors crystallize in either cubic zinc-blende or hexagonal wurtzite close-packed (hcp) structures (Mondal *et al.* 2013). ZnO is a compound consisting of interconnected zinc (Zn) and oxygen (O) atoms. ZnO has a wurtzite-type crystal structure, meaning that each zinc atom is surrounded by six oxygen atoms, forming a hexagonal structure. This arrangement in the crystal structure determines specific physical and electrical properties of ZnO (Yoon *et al.* 2015).

Under normal conditions, ZnO is an ionic semiconductor in terms of its covalent and ionic nature. In the hexagonal structure of the ZnO unit cell, each Zn atom is surrounded by four O atoms in the first shell and twelve Zn atoms in the second shell (Figure 2.3).

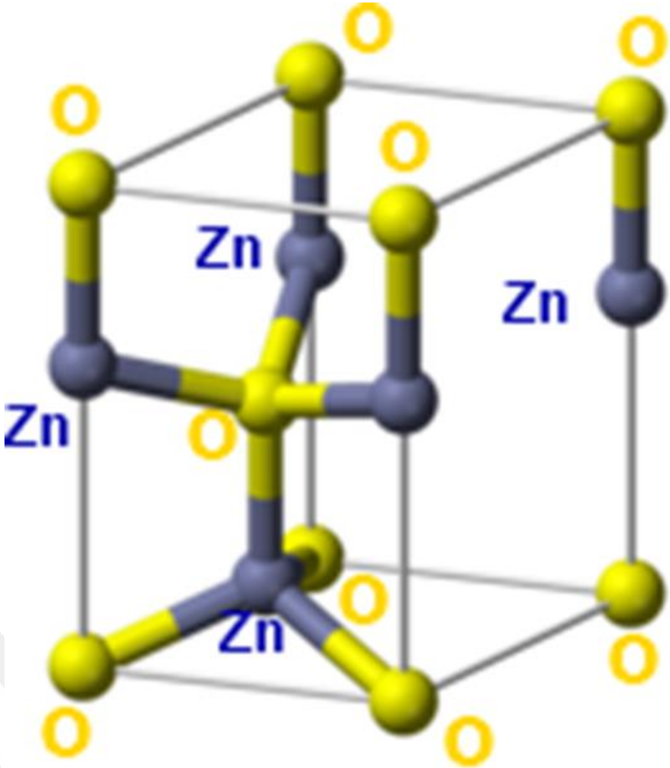

Figure 2.3 Hexagonal wurtzite crystal structure of zinc oxide (Anta *et al.* 2012)

2.4.2. Optical properties

The optical properties of ZnO are particularly notable for having a wide bandgap energy range (Zhuang *et al.* 2019). This feature allows it to efficiently absorb ultraviolet (UV) light, making it ideal for applications such as optical sensors and UV filters (Grandbois *et al.* 1999).

2.4.2. Semiconductor properties

ZnO, exhibiting high electrical conductivity, is considered a critical material in the development of semiconductor devices and innovations in nanotechnology. With various doping methods, the properties of the semiconductor can be controlled, allowing for the attainment of desired characteristics.

2.4.3. Chemical resistance

ZnO exhibits chemical resistance against many solvents (Taylor 1964). This property enables the material to be used in various industrial processes and applications.

2.4.4. Thermal stability

ZnO can remain stable even at high temperatures, allowing it to be used in high-performance electronic devices and thermally challenging environments. These characteristics of ZnO enable its wide application in various fields, including solar energy conversion, LED technology, nanotechnology-based devices, environmental sensors, medical devices, and antibacterial coatings.

2.5. Thin Film Fabrication Techniques

Thin films are materials applied in thin layers, typically consisting of a few atomic layers, onto the surface of a material, specifically produced for a particular application. The properties of these materials can be controlled based on their thickness, composition, and structure, which can be adjusted according to application and industry requirements. Thin films can be defined as coatings ranging in thickness from 100 Å to several µm (Das *et al.* 2018).

Thin films can grow on the target surface in three different ways: the first is island growth (Volmer and Weber, 1926), the second and most ideal growth is layer-by-layer growth (Frank and Van der Merwe, 1949), and the third type of growth is a combination of both island and layer growth, known as mixed growth (Figure 2.4).

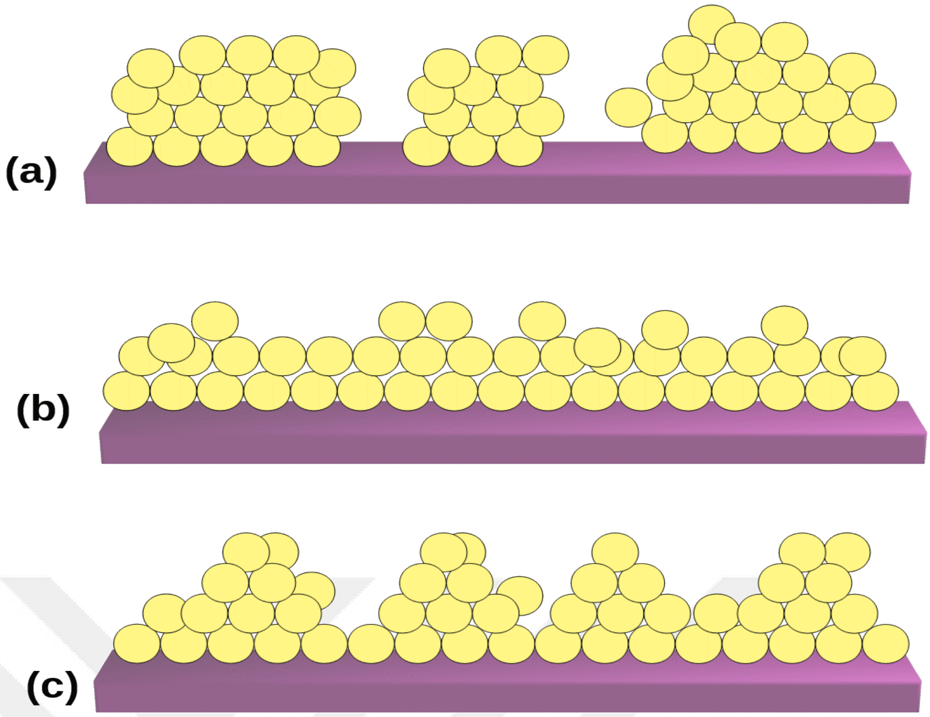

Figure 2.4 (a) Island growth, (b) Layer-by-layer growth, (c) Mixed growth (Volmer and Weber 1926)

Thin film fabrication techniques play a crucial role in determining the properties of materials and enabling their use in applications. Thin film fabrication techniques can be broadly categorized into two main types: (a) Physical methods and (b) Chemical methods (Figure 2.5).

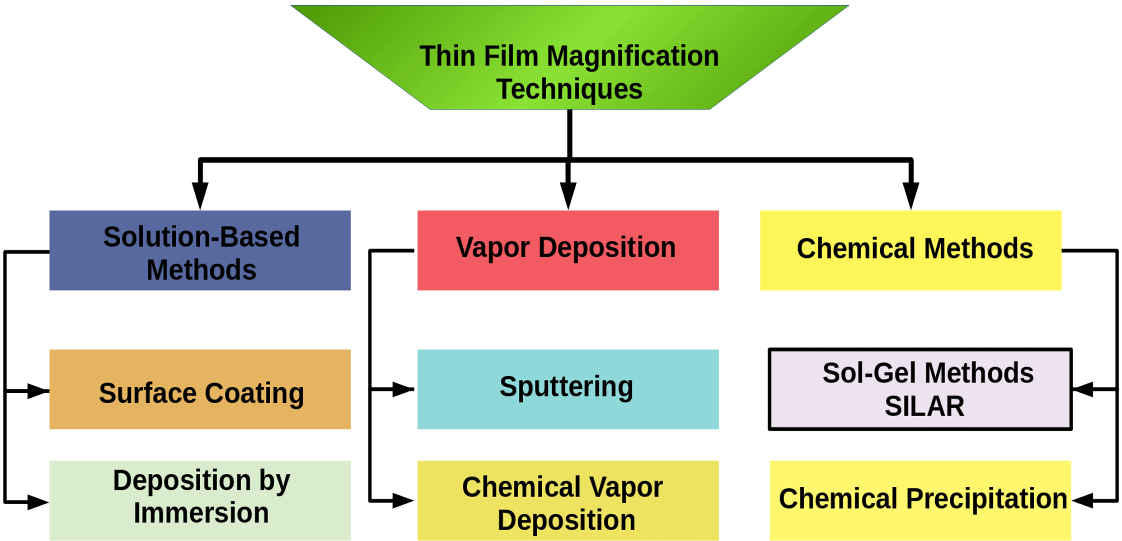

Figure 2.5 Main methods used in thin film production (Hernandez *et al.* 2008)

In this thesis, the SILAR method has been employed due to its numerous advantages, including not requiring high temperature and vacuum, being a simple method, being applicable to large surfaces, not involving complex tools and machinery in the process, ensuring uniform coating thickness throughout the coated material, creating a homogeneous structure, and not causing air pollution.

2.6. SILAR Technique

The Successive Ionic Layer Adsorption and Reaction (SILAR) technique is preferred in many applications due to its advantages such as cost-effectiveness, not requiring a vacuum environment unlike other semiconductor film growth methods, ease of controlling layer-by-layer growth (including thickness, optical, electrical, and mechanical properties), and easy applicability (Alferov et al., 1971). This method involves successive ionic layer-by-layer reactions and has found wide application in various industrial, scientific, and technological fields. SILAR method is considered ideal for obtaining high-quality doped and undoped ZnO thin films (Akman 2020).

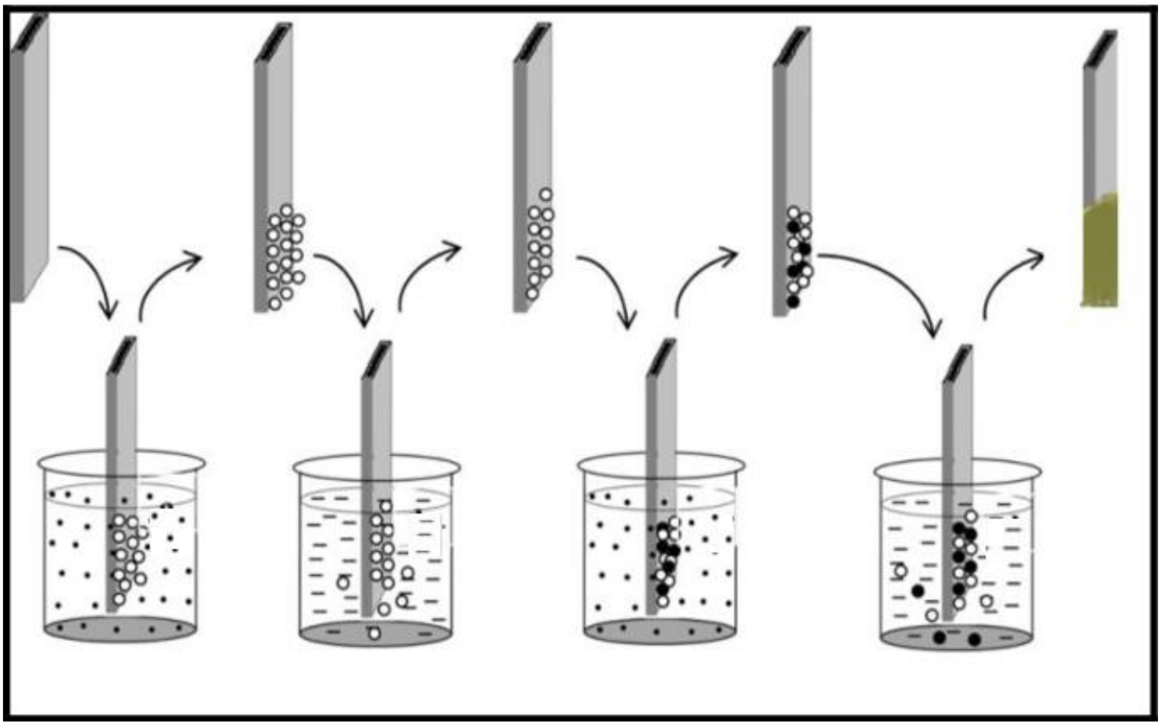

Figure 2.6 Schematic representation of the SILAR thin film growth method (Dhawankar *et al.* 2016)

SILAR is based on the principle of sequentially applying ionic solutions or gases onto a substrate, followed by the formation of a thin film layer through chemical reaction. The thickness of each layer can be controlled through step-by-step reactions. This allows for the precise management of the thin film thickness at the atomic level, enabling the production of films with desired properties. SILAR enhances film quality and helps reduce unwanted defects. Additionally, atomic-level thickness control enables the creation of very thin films. It can be used compatibly with different materials, providing a wide range of applications.

The SILAR method finds extensive use in various fields, including the production of electronic devices such as transistors, sensors, and microchips. It also has applications in the development of high-performance solar cells and optical devices.

3. MATERIALS AND METHODS

3.1 Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is a powerful imaging technique used to examine surface morphology and properties (Crewe *et al.* 1969). It scans the surface using an electron beam to obtain high-resolution images and provides detailed structural information. This technique is widely used by researchers in various disciplines.

In SEM, the electron beam is generated from an electron source, commonly utilizing thermionic or field emission methods (Zheng *et al.* 2011). Electrons are accelerated and focused under high voltage to create a high-energy beam. The sample to be examined is rendered conductive by coating it with a thin layer of gold or carbon. This step reduces electron backscattering from the sample surface, resulting in improved image quality. The sample is placed in a vacuum as the electron beam travels in a vacuum environment (Abdulrahman *et al.* 2021). The electron beam is focused and directed using magnetic or electrostatic fields, allowing it to uniformly reach the sample surface and perform the scanning process.

The electron beam scans the sample surface, generating signals such as backscattered electrons, secondary electron emissions, and secondary X-rays when electrons interact with the sample surface. These signals are captured and processed by detectors. They produce high-resolution images displaying the topographic, morphological, and compositional features of the sample surface (Figure 3.1).

The obtained images are processed and analyzed by computers. They provide information about the surface's topographic details, sample components, structural features, and other characteristics. This step offers researchers or users the necessary data to comprehend and interpret the properties of the sample. SEM is a powerful tool for observing fine details of surfaces and finds extensive applications in the examination and characterization of samples (Choy 2003).

SEM's features and advantages are composed of a series of factors, including analysis and visualization capabilities, application areas, and flexibility of use. These advantages are generally:

- SEM has the capability to observe details at the nanometer level, allowing for the observation of very small structures, textures, and morphological details on the surface of samples
- The ability to see details deep into the samples provides a three-dimensional understanding of structures
- It can work on various samples such as metals, ceramics, plastics, and semiconductors
- It can observe fine details of biological samples, including cells, tissues, and biomolecules
- It can observe the surface topography and morphology of samples in detail
- It allows for the examination of the components, chemical compositions, and distribution of samples
- Provides quick imaging and data analysis
- Can perform optimized analyses for different samples through various operating conditions and modes

SEM is utilized in various fields due to these features. It's employed for examining material structures, coatings, metallurgy, nanomaterials, analyzing cell structures, biological tissue, and investigating biomolecules in biological research. It's also used for the analysis of prehistoric and geological materials.

Figure 3.1 Hitachi SU 5000 field emission scanning electron microscope (Kokubun *et al.* 2003)

3.2 Annealing Oven

Annealing is a process that involves heating a material to a specific temperature and then cooling it slowly to make it more ductile, reduce internal stresses, and increase its strength (Roselli *et al.* 2003). Annealing ovens are used for this process, and they are commonly found in various industries such as metallurgy, electronics, and glass manufacturing. In this article, we will discuss the basic principle of operation, types, and applications of annealing ovens.

Annealing ovens come in a variety of forms, such as vacuum, continuous, and batch ovens. differed types of ovens are appropriate for varied purposes according on the material that needs to be annealed. The growth of different industries relying on this technology rely on the annealing oven's constant advancement (Figure 3.2).

Following the SILAR deposition, the ZnO thin films doped with Ni (1 %, 2 %, 3 %) on the FTO/glass substrate were annealed in a furnace at 300 °C for 15 minutes. During the annealing process, the furnace was heated to 300 °C, and the samples were placed inside. Figure 3.2 illustrates the annealing furnace.

Figure 3.2 Annealing oven (Uche 2013)

3.3. X-Ray Diffraction (XRD)

X-ay Diffraction (XRD) is a powerful analysis technique widely used for the analysis and characterization of crystal structures. It is based on the principle of X-rays being diffracted by materials to determine their atomic arrangements and crystal structures. X-rays were discovered in 1895 by the German physicist Wilhelm Röntgen. In 1912, Max von Laue and his students Friedrich and Knipping conducted X-ray diffraction experiments to understand the nature of crystal structures, studying the scattering of X-rays by crystal structures and the formation of specific patterns. This work laid the foundation for the XRD technique and generated significant interest in the scientific community.

XRD is utilized to determine the crystal structure and atomic arrangement of materials (Liu *et al.* 2021). It offers the ability to examine details such as the symmetry, cell parameters, and atomic lattices of crystal structures. XRD is employed in determining the mechanical, thermal, electrical, optical, chemical, and physical properties of materials. Fundamentally, this technique relies on the interaction and scattering of X-rays by crystal structures. Its operation occurs in the following steps:

The first step is the X-ray source. Usually, this source is in the form of a tube, which accelerates high-energy electrons toward a target to generate X-rays. These X-rays are typically generated from a specific element like copper. The second step involves sample preparation. The sample is specially prepared for analysis. Typically, a material in powder form or with a thin film layer is fixed onto a flat and clean surface.

The third step involves the interaction of X-rays with the sample. X-rays are directed towards the sample, interacting with the atomic arrangements on its surface. Atoms in the crystal structure scatter X-rays at specific angles, creating diffraction patterns. These scattering events result in diffraction patterns at various angles.

In the fourth step, the detection of diffraction patterns occurs. After the X-rays scatter from the sample, they are measured by a detector. The detector records the intensity and angular distribution of the X-rays. The fifth step involves the Bragg's Law and pattern analysis. Bragg's Law describes how scattered X-rays interfere constructively at specific angles. X-ray diffraction can be explained using Bragg's Law, represented by the simplest form of the equation in Equation (3.1) below.

$$n\lambda = 2dsin\theta \tag{3.1}$$

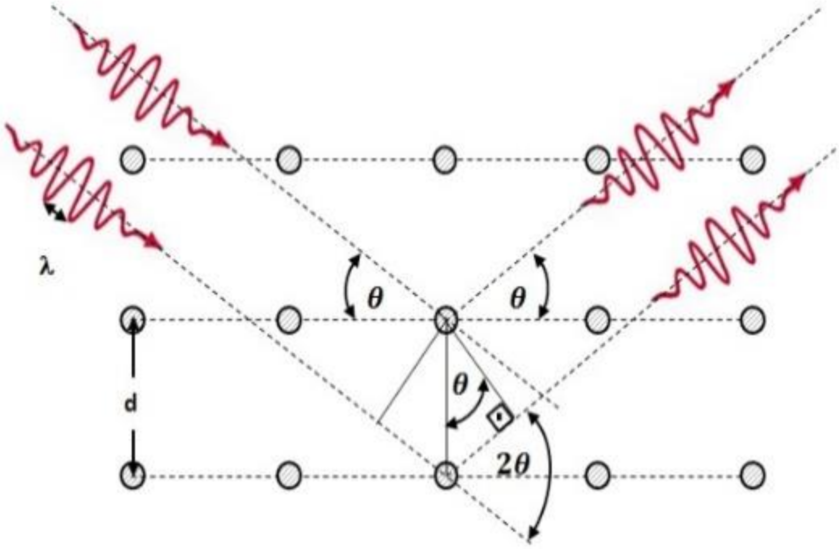

Figure 3.3 Bragg kanununun şematik gösterimi (Kitamura *et al.* 2007)

Figure 2.3 illustrates the distance d between lattice planes, the θ-Bragg angle, n as the scattering order, and λ as the wavelength. The measured diffraction patterns are analyzed and interpreted using Bragg's Law (Figure 2.3). In the final step, structural analysis occurs. Data obtained from the diffraction patterns are used to determine the parameters of the crystal structures of samples and analyze their atomic arrangements. This analysis aids in determining the crystal structures and sometimes the composition of the samples.

XRD is a powerful tool used to determine the atomic arrangements of materials (Bala *et al.* 2016). This technique plays a critical role in understanding the structural and physical properties of materials, designing new materials, and their industrial applications (Kitture *et al.* 2015). XRD is widely employed for structural analysis of semiconductors, metallurgy, ceramics, characterization of crystal structures in minerals, and determination of crystal structures in pharmaceutical molecules.

In this thesis study, the XRD analyses of the produced samples were performed using the APD 2000 PRO XRD device available at the Photonics Application and Research Laboratory of Gazi University (Figure 2.4).

Figure 3.4 APD 2000 PRO brand X-ray diffraction device (Jones *et al.* 2008)

3.4. UV-VIS Spectrometer

When ions, atoms, or molecules in a sample transition from one energy state to another, a process known as spectroscopy is used to detect and analyze the electromagnetic radiation that is emitted or absorbed is a sort of absorption spectroscopy that makes use of light from the electromagnetic radiation spectrum's neighboring visible and UV ranges. The principal absorption wavelength varies from 100 to 700 nm Figure 3.5.

Figure 3.5 UV-VIS spectrometer (Pradhan *et al.* 2015)

3.5 SILAR Method

SILAR method is an important technique used for thin film production in semiconductors (Roselli *et al.* 2003). This method is particularly advantageous in modifying the structural properties of semiconductor materials, especially their optical and electrical properties, and controlling processes. Semiconductor thin films produced by this method are preferred in many electronic, optical, and energy applications, particularly due to the controllability of layer thickness (Figure 3.3).

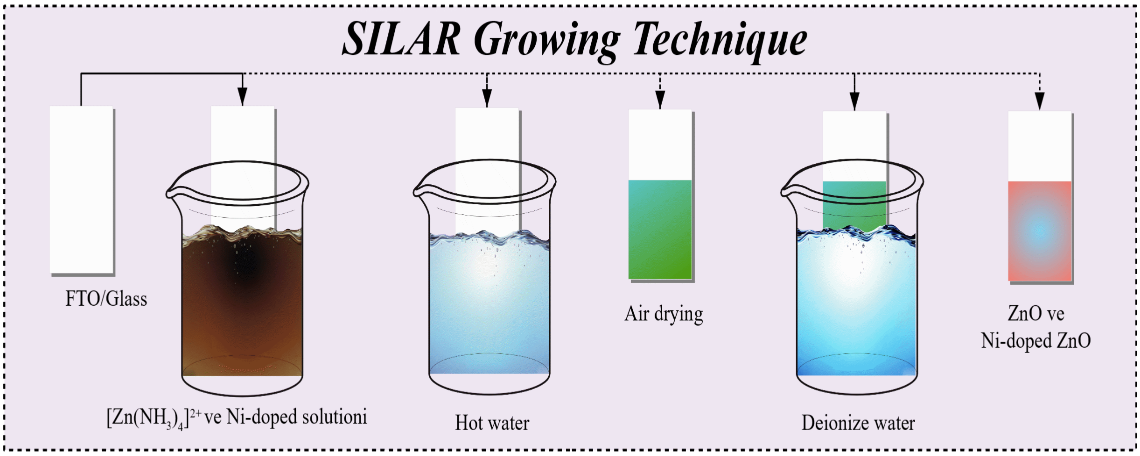

Figure 3.6 SILAR growing technique (Wasa *et al.* 2004)

In this thesis study, undoped ZnO/FTO and Ni (1 %, 2 %, 3 %) doped ZnO/FTO samples were produced using the SILAR method. The production processes have been extensively described in relevant sections.

3.4 Formation of ZnO films

Liquid ammonium is added slowly to make the 0.1 M Zn $(NO_3)_2$ solution used as a zinc source alkaline. This reaction is expressed as follows Equation (3.1).

$$Zn(NO_3)_2 + 2NH_4OH \rightarrow Zn(OH)_2 + 2NH_4^{+} + 2NO^{-} \qquad (3.2)$$

However, when enough NH_4OH is added, Zn^{+2} ions in the environment are reduced and Zn $(NH_3)_4^{+2}$ is formed. Precipitation in the solution is thereby prevented. Furthermore, the solution appears clear and transparent, Equation (3.2):

$$Zn(OH)_2 + 4NH_4^{+} \rightarrow Zn(NH_2)_4^{\ +2} + 2H_2O + 2H^{+} \qquad (3.3)$$

Glass bases, one surface of which is film-coated, are prepared for measurement Equation (3.3) and Equation (3.4):

$$H_2O_2 + H_2O \rightarrow 2OH^{+} + 2H^{+} + 1/2O_2 \qquad (3.4)$$

$$Zn(NH_3)_4^{+2} + 2OH^- \rightarrow ZnO + 4NH_3 + H_2O \quad (3.5)$$

The chemical reactions that take place during the growth stage of Ni-doped thin films are given below Equation (3.5), Equation (3.6), Equation (3.7), and Equation (3.8).

$$ZnCl_2 + 2NH_4OH \leftrightarrow Zn(OH)_2 + 2NH_4^+ + 2Cl^- \quad (3.6)$$

$$Zn(OH)_2 + 4NH_4^+ \leftrightarrow [Zn(NH_3)_4]^{2+} + 2H_2O + 2H^+ \quad (3.7)$$

$$NiCl_2 + 2NH_4OH \leftrightarrow Ni(OH)_2 + 2NH_4^+ + 2Cl^- \quad (3.8)$$

$$Ni(OH)_2 + 4NH_4^+ \leftrightarrow [Ni(NH_3)_4]^{2+} + 2H_2O + 2H^+ \quad (3.9)$$

4. RESULTS AND DISCUSSION

This section evaluated the analysis methods and results of undoped ZnO/FTO and (ZnO/FTO/Ni 1 %, ZnO/FTO/Ni 2 %, and ZnO/FTO/Ni 3 %) samples grown using the SILAR technique. XRD, SEM-EDX, and optical absorption analyses were performed on the undoped ZnO/FTO and (ZnO/FTO/Ni % 1, ZnO/FTO/Ni % 2, and ZnO/FTO/Ni % 3) samples, and the outcomes were interpreted.

4.1 Morphological and Elemental Analysis of Thin Films

4.1.1. SEM Analysis

Analyses of the thin films produced based on the doping ratio of undoped ZnO/FTO and (ZnO/FTO/Ni 1 %, ZnO/FTO/Ni 2 %, and ZnO/FTO/Ni 3 %) samples were sequentially conducted. SEM analysis was performed to examine the morphology of the produced semiconductor thin film samples in detail. The undoped ZnO/FTO samples exhibited large clusters on the surface. In contrast, Ni-doped samples displayed sparser and smaller clusters on the FTO surface. An increasing trend in cluster formation on the surface was observed as the Ni doping increased. A more homogeneous distribution on the surface was observed in the ZnO/FTO/Ni 3 % sample. SEM images of the produced samples are presented in Figure 4.1.

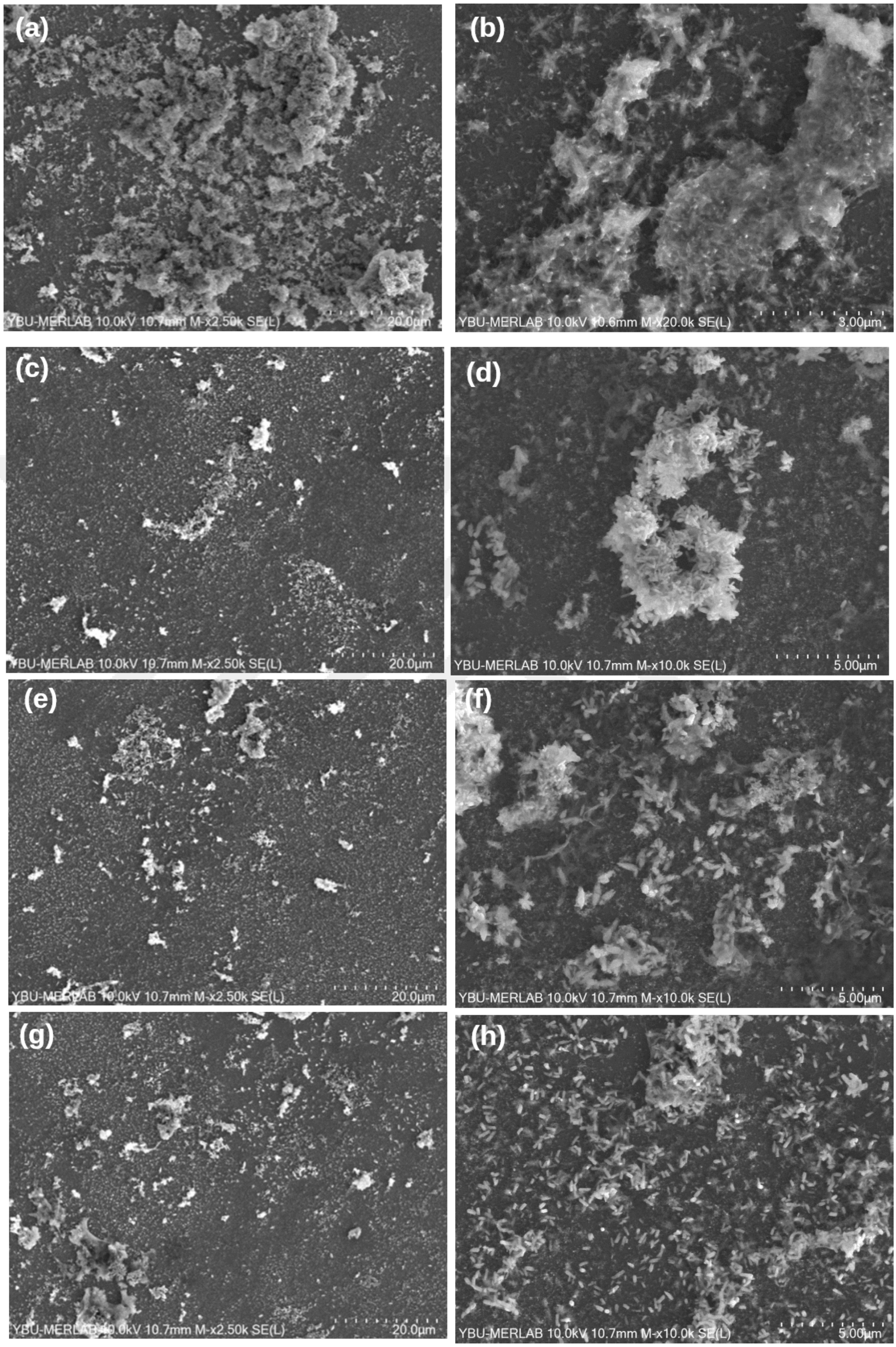

Figure 4.1 SEM analysis of thin films of (a) FTO/ZnO, (b) FTO/ZnO/Ni 1 %, (c) FTO/ZnO/Ni 2 %, (d) FTO/ZnO/Ni 3 %

4.1.2. EDX analysis

EDX measurements were conducted to understand the elemental contributions and resulting compositional ratios of the produced undoped ZnO/FTO and (ZnO/FTO/Ni 1%, ZnO/FTO/Ni 2%, and ZnO/FTO/Ni 3%) samples. The images of these measurements are provided in Figure 4.2.

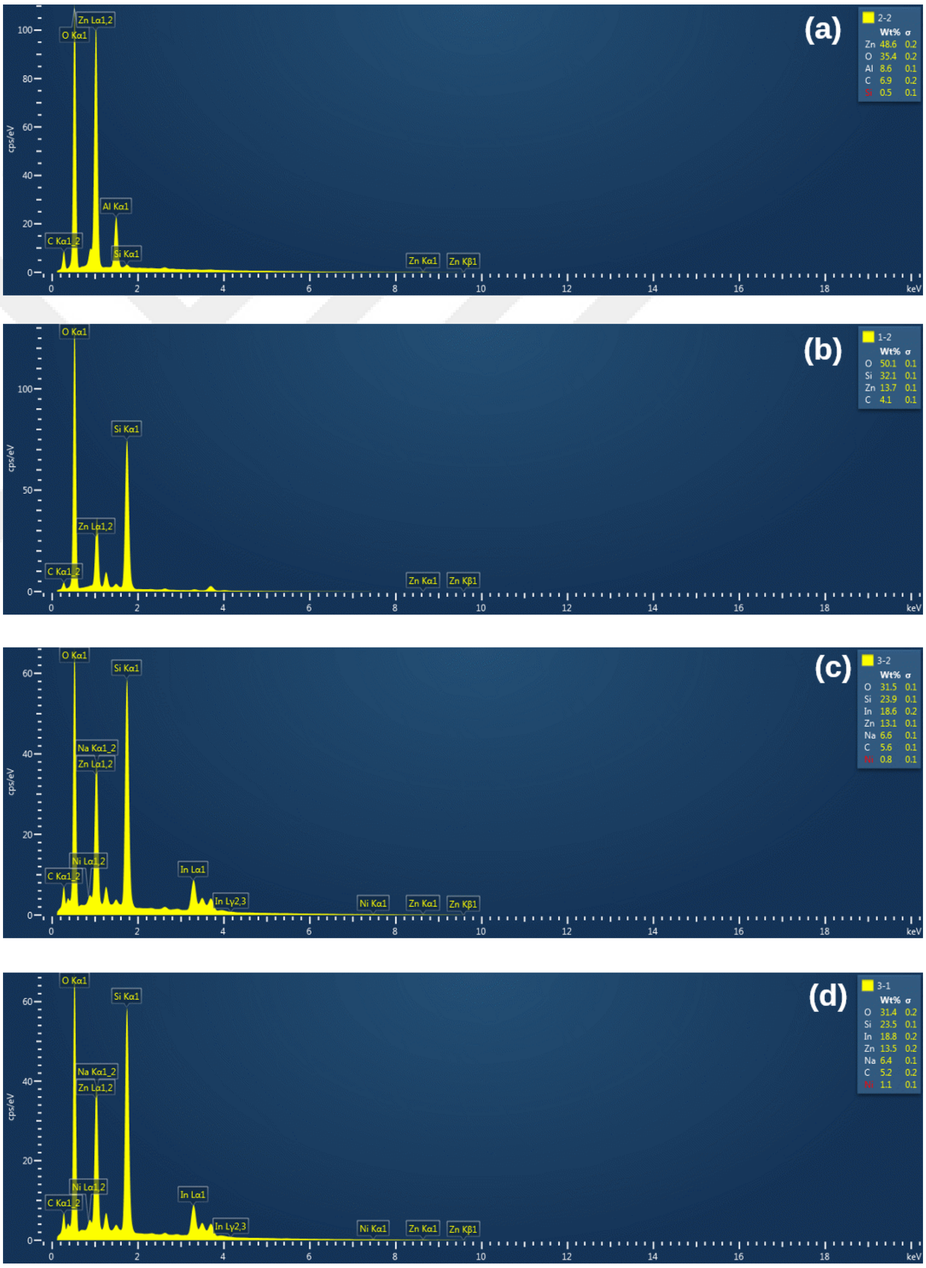

Figure 4.2 EDX analysis of thin films of (a) FTO/ZnO, (b) FTO/ZnO/Ni 1 %, (c) FTO/ZnO/Ni 2 %, (d) FTO/ZnO/Ni 3 %

The presence of Si, O, C, and Na elements can be observed in the EDS spectra of the examined samples. The existence of Si originates from precursor materials used in the synthesis process and from the glass substrate. The Na element, on the other hand, is an unwanted impurity introduced during the synthesis of thin films. It was noticed that as the Ni doping ratio increased, there was an increase in the detected peak intensity in the EDS spectra. Unexpectedly, the presence of the In element has been observed in the EDS analysis. This occurrence may indicate potential contamination of the sample. The presence of the In element suggests that the sample may have been affected by external factors during the preparation process, which could influence the analysis results.

4.2 XRD Analizleri

Figure 4.3 presents the XRD analyses of the produced ZnO/FTO and (ZnO/FTO/Ni % 1, ZnO/FTO/Ni % 2, and ZnO/FTO/Ni % 3) samples.

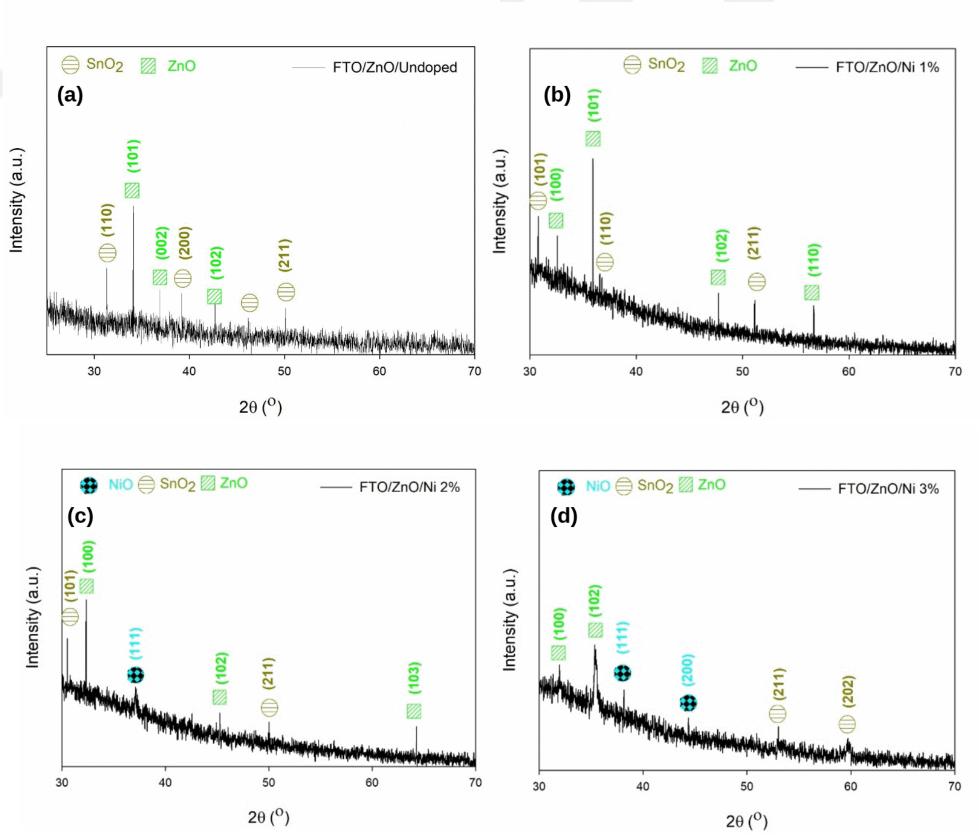

Figure 4.3 XRD analysis of thin films of (a) FTO/ZnO, (b) FTO/ZnO/Ni 1 %, (c) FTO/ZnO/Ni 2 %, (d) FTO/ZnO/Ni 3 %

The dominant diffraction peaks correspond to FTO based on the (101), (202), and (211) orientations, indicating a tetragonal crystal structure. As the Ni-doped concentration increases, there is an increase in peak intensities and a decrease in the half-width of the peaks in the films. Additionally, it's observed that with an increase in doping concentration, compared to undoped films (ZnO), there's a decrease in peak intensities and an increase in the half-width of the peaks. When examining the crystallization levels of the films, the FTO/ZnO/Ni 3 % sample exhibited the best conformity.

In the investigated films, the binary phase of NiO and ZnO has been observed, indicating the presence of both NiO and ZnO crystal structures in these films (Figure 4.3 c, and Figure 4.3 d). Such a structure can influence the physical and optical properties of the material and is crucial for understanding how the material behaves in specific applications.

4.3 Optic Analysis

The absorption spectra drawn using the absorption measurements taken at room temperature for the produced undoped ZnO/FTO and (ZnO/FTO/Ni 1 %, ZnO/FTO/Ni 2 %, and ZnO/FTO/Ni 3 %) samples grown via the SILAR method are presented in Figure 3.3. The calculated forbidden energy bandgaps (E_g) of the films were obtained by plotting $(\alpha h\nu)^2$ $(eVcm^{-1})^2$ as a function of energy in Figure 3.4.

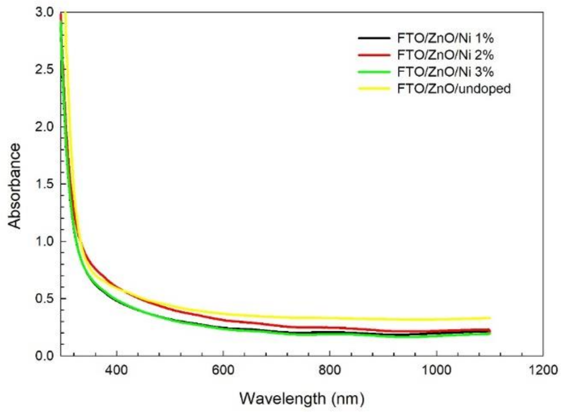

Figure 4.4 Energy dependent plots of $(\alpha h\nu)^2$ $(eVcm^{-1})^2$ plotted using optical absorption measurements of FTO/ZnO/undoped, FTO/ZnO/Ni 1 %, FTO/ZnO/Ni 2 %, FTO/ZnO/Ni 3 % thin films

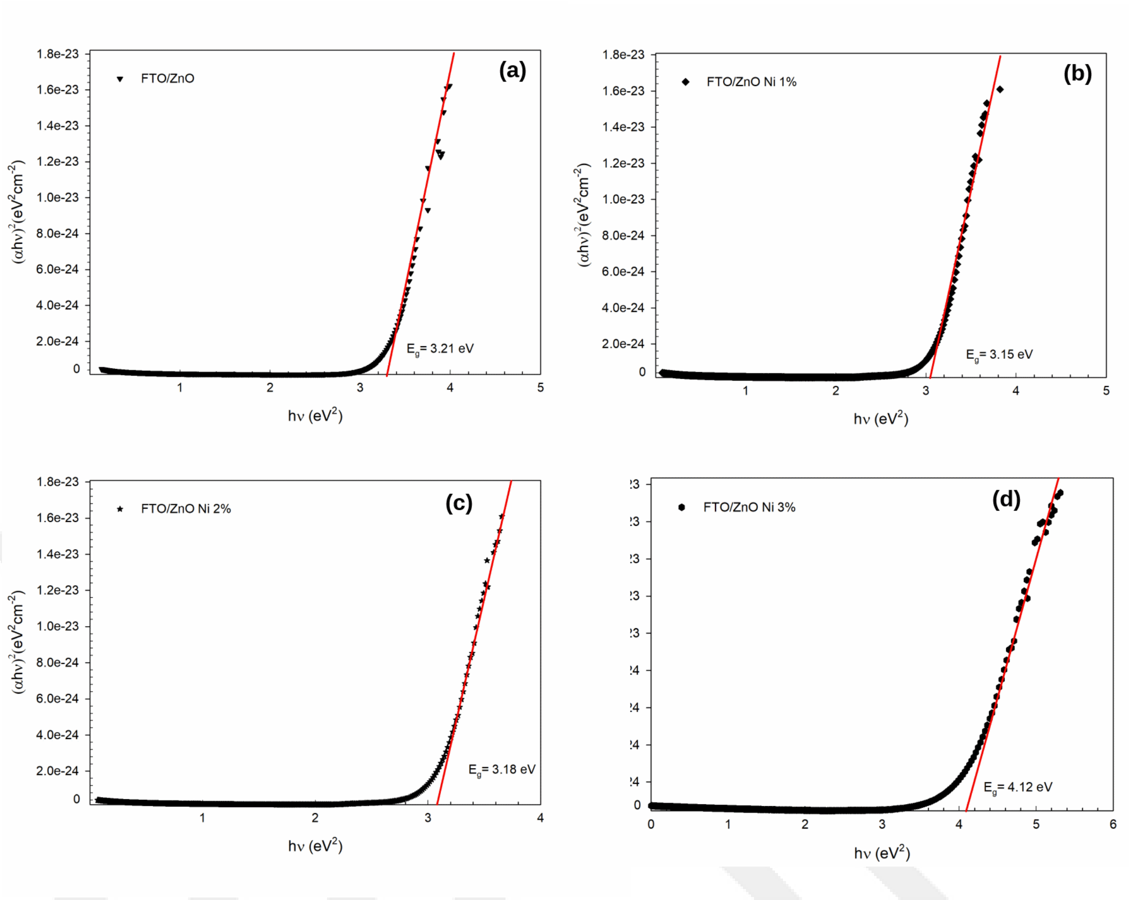

Figure 4.5 Energy dependent graphs of $(\alpha h\nu)^2$ $(eVcm^{-1})^2$ plotted using optical transmittance measurements of FTO/ZnO, FTO/ZnO/Ni 1 %, FTO/ZnO/Ni 2 %, FTO/ZnO/Ni 3 % thin films

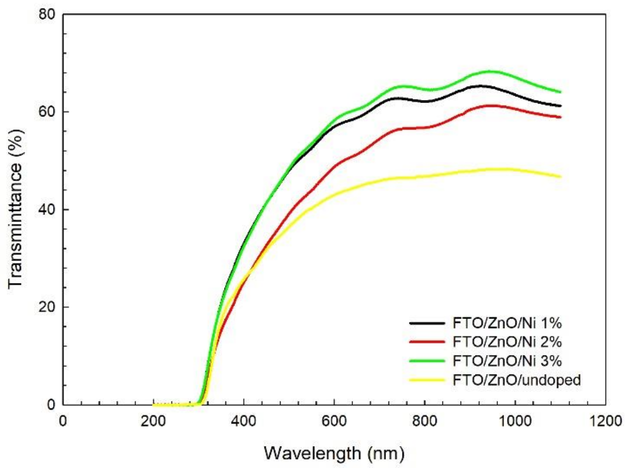

Figure 4.6 Energy-dependent band gap graphs of $(\alpha h\nu)^2$ $(eVcm^{-1})^2$ plotted using optical absorption measurements of (a) FTO/ZnO, (b) FTO/ZnO/Ni 1 %, (c) FTO/ZnO/Ni 2 %, (d) FTO/ZnO/Ni 3 % thin films

Table 4.1 presents the calculated forbidden energy values of FTO/ZnO, FTO/ZnO/Ni 1%, FTO/ZnO/Ni 2%, and FTO/ZnO/Ni 3% thin films derived from the graphs of $(eVcm\text{-}1)^2$ vs. hυ (energy). The altered doping levels reveal variations in the forbidden energy range of the manufactured thin films. The thin films doped with Ni exhibit a reduction in their forbidden energy ranges, leading to a more distinct absorption edge and increased absorption intensity. The decrease in the forbidden energy range due to Ni doping can be associated with the enhancement of crystalline structure and surface properties of the generated thin film, coupled with an increase in grain size

Table 4.1 The forbidden energy (E_g) and Urbach (E_u) values calculated from the energy E_g) and Urbach (Eu) values calculated from the energy dependent graphs of $(\alpha h\nu)^2$ $(eVcm^{-1})^2$ drawn using optical absorption measurements of FTO/ZnO/Ni 1 %, FTO/ZnO/Ni 2 %, FTO/ZnO/Ni 3 % thin films

Sample	**E_g (eV)**	**E_u (meV)**
FTO/ZnO	3.21	292.55
FTO/ZnO/Ni 1 %	3.15	328.49
FTO/ZnO/Ni 2 %	3.18	333.17
FTO/ZnO/Ni 3 %	4.12	451.14

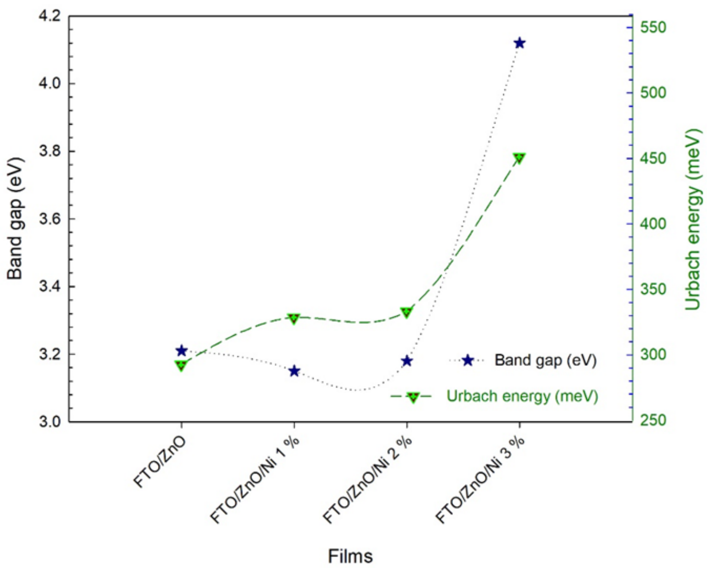

Figure 4.7 Band gap and Urbach energy versus of FTO/ZnO/Ni 1 %, FTO/ZnO/Ni 2 %, FTO/ZnO/Ni 3 %

5. CONCLUSION AND RECOMMENDATIONS

Increasing energy demand and environmental concerns have driven a growing interest in sustainable and renewable energy sources. In this context, thin film-based solar cell technologies, gas sensors, and biological sensors have become areas of intensive research due to their high performance and cost-effectiveness. Materials based on ZnO play a significant role in this field; these films constitute a fundamental component of photovoltaic devices owing to their high conductivity and optical transparency.

Studies investigating the impact of dopants on the properties of ZnO semiconductor films are crucial for further enhancing their characteristics and improving their efficiency. In this regard, this thesis delves deeply into the structural, surface, and optical properties of Ni-doped ZnO thin films produced via the SILAR method.

The XRD analysis of ZnO films produced with different ratios of Ni doping, ranging from pure ZnO to 1%, 2%, and 3% Ni doping, revealed an increase in peak intensity and a decrease in full width at half maximum (FWHM) with higher Ni doping levels. Moreover, an increase in the doping ratio (x) resulted in decreased peak intensity and increased FWHM compared to undoped ZnO films. The dominant diffraction peaks were identified as oriented towards the tetragonal crystal structure of SnO2:(FTO), namely (101), (202), and (211). Additionally, as the Ni-doped concentration increased, the films exhibited increased peak intensity and decreased FWHM. Evaluation of these results suggests that the crystallization levels of the produced thin films, upon closer examination, indicate the best alignment in the FTO/ZnO/Ni 3% sample

When examining the morphology of the thin film samples, it was observed that undoped ZnO/FTO samples formed large clusters on the surface. In contrast, Ni-doped samples exhibited sparser, smaller clusters on the FTO surface. An increasing Ni-doping ratio showed an upward trend in the formation of surface clusters. Additionally, a more homogeneous distribution on the surface was observed in the ZnO/FTO/Ni 3% sample among the produced samples.

Through the optical absorption measurements of the produced ZnO films, a decrease in the bandgap energy values of the films was observed with an increase in the doping ratio. The decrease in the bandgap energy with increasing doping showed a reduction from ZnO (room temperature undoped Eg = 3.21 eV) to (room temperature E_g = 3.12 eV - 3.18 eV). The bandgap energy values of the thin films produced with Ni doping decreased, resulting in a sharper absorption edge and increased absorption intensity.

The outcomes obtained from this thesis indicate that Ni-doped doping is a significant variable in the production of ZnO thin films, whether undoped or produced with 1%, 2%, and 3% Ni doping ratios. These findings have the potential to form a basis for advanced research in materials science and semiconductor-based gas sensor technologies, among other areas.

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