ELECTRICAL CHARACTERIZATION OF VANADIUM (VO₂) THIN FILMS GROWN BY MAGNETRON SPUTTERING TECHNIQUE

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ABSTRACT

ELECTRICAL CHARACTERIZATION OF VANADIUM DIOXIDE (VO2) THIN FILMS GROWN BY MAGNETRON SPUTTERING TECHNIQUE

The aim of this thesis is to determine the parameters required for the production of pure VO₂ thin films and to evaluate the usability of these films by calculating their MIT ratios. Vanadium dioxide (VO₂), a substance that exhibits a phase transition from the insulating state to the metallic state at approximately 68 °C, shows significant changes in its electrical conductivity and optical properties. To get pure VO₂ thin films, and get optimum parameters for deposition, several parameters were varied during the manufacturing process, including temperature, oxygen-argon ratio, and coating time. Then, electrical characterization of the produced films was performed. Examining the temperature dependence of the resistance of thin films has been a critical aspect of electrical measurements to determine the MIT transition. With the help of the probe station, it was determined that the MIT transition occurred at approximately 68 °C. The results obtained using structural characterization techniques revealed that characteristic vibration modes were observed in the analyses performed with Raman spectroscopy, while XRD analyses revealed that the crystal structure was preserved. Additionally, XPS analyses did not detect any surface contamination other than carbon. SEM and EDX analyses evaluated the surface morphology and elemental stoichiometry and showed that the internal structure of the films was intact. These results support the usability of VO₂ thin films in advanced electronic and optical applications and contribute to the determination of optimum production parameters. This thesis demonstrates the potential of VO₂ in various technological applications by efficiently utilizing MIT properties.

ÖZET

MIKNATISSAL SAÇTIRMA TEKNİĞİ İLE BÜYÜTÜLEN VANADYUM DİOKSİT (VO2) İNCE FİLMLERİN ELEKTRİKSEL KARAKTERİZASYONU

Bu tezin amacı, saf VO2 ince filmlerin üretimi için gerekli parametreleri filmlerin MIT oranlarını hesaplayarak kullanılabilirliğini belirlemek ve bu değerlendirmektir. Yaklaşık 68 °C'de yalıtkan durumdan metalik duruma faz geçişi sergileyen bir madde olan vanadyum dioksit (VO2), elektriksel iletkenliğinde ve optik özelliklerinde önemli değişiklikler gösterir. Üretim süreci sırasında sıcaklık da dahil olmak üzere oksijen-argon oranı ve kaplama süresi gibi bir dizi parametre değiştirildi. Daha sonra üretilen filmlerin elektriksel karakterizasyonu yapıldı. İnce filmlerin direncinin sıcaklığa bağımlılığının incelenmesi ve MIT geçişini belirlemek için elektriksel ölçümlerin kritik bir önemi olmuştur. Prob istasyonunun yardımıyla MIT geçişinin yaklaşık 68 °C'de gerçekleştiği tespit edildi. Yapısal karakterizasyon teknikleri kullanılarak elde edilen sonuçlar, Raman spektroskopisi ile yapılan analizlerde karakteristik titreşim modlarının gözlemlendiğini, XRD analizleri ise kristal yapının korunduğunu ortaya koymuştur. Ek olarak, XPS analizi karbon dışında herhangi bir yüzey kirliliği tespit etmemiştir. SEM ve EDS analizleri yüzey morfolojisini ve elementel stokiyometriyi değerlendirmiş ve filmlerin iç yapısının sağlam olduğunu göstermiştir. Bu sonuçlar VO2 ince filmlerinin ileri elektronik ve optik uygulamalarda kullanılabilirliğini desteklemekte optimum üretim parametrelerinin ve belirlenmesine katkıda bulunmaktadır. Bu tez, MIT özelliklerini verimli bir şekilde kullanarak VO2'nin çeşitli teknolojik uygulamalardaki potansiyelini göstermektedir.

Mon amour, mon ami.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

The metal-insulator transition (MIT) in certain materials refers to a phenomenon where they undergo a transition from a metallic (conductive) state to an insulating (nonconductive) state under specific conditions such as temperature, pressure, or external stimuli. This transition can have significant implications for the material's electrical, optical, and thermal properties. Vanadium dioxide (VO₂) belongs to a group of transition metal oxides exhibiting metal-insulator transitions (MIT) near room temperature at T_{MIT} \approx 68 °C (Yuce, 2015). Vanadium dioxide displays a critical temperature of around 68 °C for MIT transition. Above the critical temperature, a structural transition to the rutile phase occurs in the material, and below this temperature, a monoclinic structure forms (Liu et al., 2018). The structural shift as a result of rearrangement in the band structure of VO₂ results in a significant alteration in electronic conductivity (Lu et al., 2021). VO₂ undergoes transition, firstly documented by Morin in 1959, from metal to an insulator when exposed to heat (Wang et al., 2019). While there are approximately 20 stable vanadium oxides, three of the specimens exhibit the transition effect from metal to semiconductor at different T_c , i.e. VO₂ at 68 °C, V₂O₅ at 257 °C, and V₂O₃ at -123 °C (Krammer et al., 2016; Pergament et al., 2013). Vanadium dioxide shows distinctive properties compared to other vanadium oxide phases, particularly the metal-insulator transition occurring at ~68 °C, near room temperature. This unique characteristic has made VO₂ the subject of extensive contemporary and theoretical research. The transition from the insulator to the metallic phase leads to a significant decrease in the resistivity of VO₂. The higher temperature of VO₂ from critical temperature corresponds to the metallic phase, while the lower temperature of T_c indicates the insulating phase. Vanadium dioxide (VO₂) films have been extensively researched for their thermochromic and thermotropic characteristics, holding significant promise for practical uses such as advanced coatings for "smart" windows (Granqvist 1990, Kamalisarvestani et al., 2013), switchable absorbers in solar thermal collectors (Paone, 2013), rapid electronic switches (Vitale et

al., 2015) and even in military contexts to thwart infrared detection (Moffat et al., 2005). Thanks to IMT property and its low 1/f noise and resistivity, it can be manufactured in a more cost-effective manner. VO₂ is preferred for use in bolometers (Alaboz, 2017), nanowires (Sun, 2011), and metamaterials in THz applications for medical imaging (D'Arco et al., 2020), wireless communication (Burla et al., 2019), etc.

1.2 Phase Transition of VO₂

VO₂ is one of the compounds that contain both a transition metal and an oxide component. It exhibits a semiconductor-metal transition at room temperature. The critical transition temperature is 68 °C, and below this temperature, the compound is monoclinic. Above 68 °C, it transforms into a rutile phase (Liu et al., 2018). This structural change results in a significant change in electronic conductivity due to changes in the band structure of VO₂ (Lu et al., 2021). VO₂ undergoes a metal-to-insulator transition when exposed to heat and this phenomenon was discovered by Morin in 1959 (Wang et al., 2019). It is estimated that there are about 20 stable vanadium oxide compounds, but only three of them exhibit semiconductor-to-metal transition behavior (Krammer et al., 2016). These compounds are VO₂, V₂O₃ and V₂O₅, have different critical temperatures for phase transition i.e. VO₂ at 68 °C, V₂O₅ at 257 °C, and V₂O₃ at -123 °C (Krammer et al., 2016; Pergament et al., 2013). Vanadium dioxide has exceptional properties compared to other vanadium oxides, such as MIT, which occurs at about 68 °C, close to room temperature.



Figure 1.1 a) Monoclinic structure and band gap b) Rutile structure and band gap (Wegkamp and Stahler, 2015). c) Graph of MIT (Wu et al., 2013).

The nature of VO_2 has been the subject of intensive experimental and theoretical research, with the objective of elucidating its properties. At the transition temperature, there is a notable reduction in the resistivity of VO_2 . A temperature above the transition temperature indicates the metallic phase of VO_2 , while a temperature below the transition temperature indicates the insulating phase. Despite the ongoing controversy surrounding the VO_2 phase transition mechanism, scientists have made significant strides in

understanding the MIT mechanism in recent years. Two theoretical explanations have been proposed for the metal-insulator transition (MIT) behavior of vanadium dioxide: the structure-based Peierls transition mechanism and the Mott-Hubbard electron correlation mechanism. The structure-based Peierls transition mechanism is founded upon transformation in lattice symmetry, whereas the Mott-Hubbard electron correlation mechanism is predicated upon an electronic transition. As illustrated in Figure 1.1, alterations in the crystal parameters occur during the phase transition, resulting in a divergence of the V atom from the vertex angle (Wegkamp and Stahler, 2015). This results in the formation of a monoclinic crystal structure with low symmetry, thereby enabling the formation of V-V bonds of varying lengths. The formation of a V-V dimer results in a direct transition from a delocalized to a localized state. In the 1930s, Rudolf Peierls proposed that one-dimensional conductors consist of equally spaced atoms, each carrying a conduction electron. This was based on the assumption that the lattice constant (a) is constant. The 1D system becomes unstable due to the interaction of electrons with the lattice, leading to lattice distortion of pairs of atoms at low temperatures. When the bandgap is opened, energy is lost as more energy is expended than the elasticity of the lattice distortion allows. In this instance, the total energy of the electronic network system is reduced, resulting in a transition. The Mott-Hubbard mechanism posits that the MIT phase transition is accompanied by a change in the band structure. The metallic behavior is identified by the position of the Fermi level between the π^* and d|| bands. However, at lower temperatures, when VO₂ adopts a monoclinic band structure, a single energy band splits into two distinct bands (d|| band and d^* || band), resulting in the formation of a bandgap between them (Shao et al., 2018). Vanadium dioxide phases can contain both insulating monoclinic (M1) and conducting rutile (R) but can also have intermediate insulating phases such as M2, M3 (monoclinic), and T (triclinic). These vanadium phases can form under pressure or lattice tension, depending on conditions (Otto et al., 2019). The most important phase of these intermediate states is M2, where half of the vanadium chains dimerize (Lu et al., 2021). Raman spectroscopy is employed to discern the various phases of VO₂ (Shi et al., 2021). Nevertheless, the Peierls mechanism is unable to account for two significant issues. Firstly, the model is unable to account for the significant band gap observed in the monoclinic phase of vanadium dioxide, which is estimated to be approximately 0.6 eV. Secondly, the Peierls mechanism is unable to account for the formation of the intermediate phase of VO₂, as evidenced by the findings of Aetukuri et al. (2013) and Shao et al. The Mott-Hubbard mechanism posits that lattice disruption is a

necessary condition for MIT, but it is not a sufficient explanation for the metal-insulator transition (Shao et al., 2018). Calculations performed by Wentzcovitch using density functional theory (DFT) identified a structure that exhibited characteristics similar to the M1 phase. However, the establishment of a band gap (with -0.004 eV) (Tiwari, 2015) was not possible. The absence of a band gap precludes the prediction of a band gap in the M1 phase and the explanation of the insulating properties of the M2 phase.

1.3 Triggering Methods for Phase Transition of VO₂

The transition of VO_2 from the insulating state to the metal state can be initiated by several different triggering methods. The aforementioned methods include UV triggering, electrical triggering, thermal triggering, and voltage triggering. Each method employs distinct physical mechanisms to initiate the metal-insulator transition (MIT) of VO_2 , with the potential for controlling the optical, electrical, and thermal properties of the material.

1.3.1 UV- Triggering

The activation energy required for the creation of oxygen sources in VO₂ was calculated to be between 3 and 3.5 eV (Zhang et al., 2016). Therefore, UV rays with a photon energy of 3.35 eV (375 nm) can generate oxygen formations in the crystal lattice of an oxygen-deficient treated VO₂ film by releasing oxygen. However, red and green light, due to their lower photon energy, cannot release oxygen from the lattice, regardless of the section. The removal of oxygen results in V atoms selecting a few electrons and transitioning to the neighboring V-3d states of these electrons. These electrons completely fill the d|| and π^* orbitals and transition to an electronic phase. Furthermore, changes in the ionic radius V due to oxygen sources and electron release in the lattice create a voltage in VO₂. This stress induces a transformation of VO₂ from a monoclinic phase to a rutile phase, thereby resulting in a metallic phase (Li et al., 2022).

1.3.2 Electrical Triggering

The electric field applied to the VO_2 thin film results in electron-electron interactions. The Mott-MIT principle posits that the applied electric field causes electrons to become mobile, thereby stabilizing the metallic phase and facilitating a metal-insulator transition. This process is solely dependent on electrical interactions. Moreover, the electric field induces electron-phonon interaction, which results in an elevated temperature of the material. As the temperature rises above 68 °C, the monoclinic lattice structure of the VO₂ thin film undergoes a transformation into a rutile structure, accompanied by a reduction in the thin film's resistivity. The electric field required for MIT to occur is dependent on a number of variables, including the structure of the device to be produced, the thickness of the thin film, and the presence of thermal support. E-MIT plays a pivotal role in regulating the resistance and performance of VO₂-based electronic devices by facilitating rapid and precise phase transitions. The following section will present a summary of the aforementioned variables (Leroy et al., 2012).

1.3.3 Thermal Triggering

 VO_2 thin film, which exhibits a transition from the insulating phase to the metallic phase at a temperature of 68 °C and above. Furthermore, the material can be heated manually through the application of thermal energy. The application of UV light and electrical triggering can result in a change in the band gap, as well as the generation of heat through electron-phonon interaction. This can subsequently induce a phase change in the VO_2 thin film above 68 °C. The thin film can be heated using thermal triggering, such as a laser or heater, which causes a change in the VO_2 lattice structure from monoclinic to rutile phase. The electrical and optical properties of the thin film are provided. It is important to exercise caution to avoid burning the film. Furthermore, it is essential to implement controlled heating processes before the factors that allow the film to heat up and exceed the critical temperature are triggered. Thermal triggering can be achieved inexpensively using many different techniques such as laser, electrical welding, and heating.

1.3.4 Strain Triggering

Vanadium dioxide (VO₂) is a strongly correlated electron material (CEM) that exhibits a metal-insulator transition (MIT) at specific temperatures. This transition results in significant alterations in the electrical conductivity and structural phase of the material (Cao et al., 2009). As VO₂ transitions from the metal phase to the insulator phase, this change can be controlled by the effects of temperature and strain (Kim et al., 2021). The structural changes that occur during the phase change of VO₂ can be manipulated by the applied strain. In particular, axial compression or stretching can affect the phase state of the material, thereby adjusting the electronic and optical properties of the material.

1.4 Application

 VO_2 thin films behave as insulators below the critical temperature of 68 °C, and above it, they transform into a metallic thin film. This property gained electrical and optical properties by controlling temperature, VO_2 thin film can used as coatings for "smart" windows (Granqvist et al., 1990, Kamalisarvestani et al., 2013), switchable absorbers in solar thermal collectors (Paone et al., 2013), rapid electronic switches (Vitale et al., 2015) and even in military contexts to thwart infrared detection (Moffat et al., 2005).

1.4.1 VO₂ for Smart Windows

Vanadium dioxide (VO₂) has garnered interest as a potential material for smart glasses due to its reversible metal-insulator transition at 68 °C. This transition involves a structural shift from the high-temperature rutile (R) phase to the low-temperature monoclinic (M) phase, along with changes in infrared optical properties. In the rutile phase, vanadium atoms are positioned at the lattice points of a body-centered cubic structure and reside at the center of VO₆ octahedra. During the phase transition from R to M (cooling mode), vanadium atoms move along the V-V direction, leading to the binding and bending of V⁴⁺ ions and the distortion of VO₆ octahedral cages (Guan et al., 2019). This structural change leads to significant changes in the optical properties of VO₂. In the

high-temperature rutile (R) phase, VO₂ exhibits metallic properties and its transmittance decreases by reflecting light in the near-infrared. In the low-temperature monoclinic phase (M), VO₂ exhibits insulating properties and transmits more near-infrared light in this phase. This increases the material's ability to reflect sunlight and dissipate heat while making it less reflective and more translucent at low temperatures (Cui et al., 2018). During the phase transition of VO_2 , light transmission in the visible range also decreases. When in the metallic phase at high temperatures, VO₂ becomes opaque in the visible and transmits less light. When it is in the insulating phase at low temperatures, the material becomes more transparent, and its light transmission increases. These optical changes also produce changes in the color of VO₂; while the material appears darker and more reflective in the metallic phase, it appears lighter and more transparent in the insulator phase. In addition, in the metallic phase, the ability of VO₂ to reflect light increases, and the material appears brighter. In the insulator phase, it absorbs more light and reflects less. These optical changes occur concomitantly with alterations in the magnetic, thermal, and conductivity properties of VO₂, making it an attractive material for smart glass applications (Granqvist et al., 1990). VO₂ plays a significant role in optimizing the thermal management of buildings by providing the ability to control the light transmittance of the glass, especially as the temperature changes.

1.4.2 Rapid Electronic Switch

The ability of VO_2 to induce metal-insulator transitions (MIT) through nonthermal stimuli such as strain, UV light, and electrical input expands its potential for various technological applications. Notably, the ultrafast switching times, in the nanosecond range, observed for electrically induced metal-insulator transitions (E-MIT) present promising opportunities for advancements in oxide electronics. (Vitale et al., 2015). While E-MIT can only occur with electrical properties due to the Mott-electronelectron interaction as described in section 1.4.1, E-MIT can cause a transition from the insulating to the metallic phase due to the Pierls transition (Pergament et al., 2015). The change in resistance makes it possible to control the current flowing through the circuit. By changing the base from which VO_2 is generated (Vitale et al., 2015), it is possible to increase the resistance of the device.

1.4.3 THz modulation

Terahertz (THz) waves occupy a pivotal position within the electromagnetic spectrum, carrying information that is essential for the development of advanced technologies such as signal sensing, wireless communications, security imaging, and more. THz waves are particularly well-suited to the demands of wireless communications. The advancement of THz technology is contingent upon the development of efficient components, including, dynamic polarization controllers, switchable mirrors, and phase modulators. In this context, the insulator-metal phase transition of VO₂ affects the transmission and reflection of THz waves, providing a promising solution for THz modulation. The phase of THz waves can be controlled by VO₂ thin films in combination with lasers or thermal sources. This characteristic renders VO₂ an optimal candidate for use as a THz modulator, playing a pivotal role in the advancement of THz technologies. In order to elucidate the methodologies employed in the utilization of VO₂ thin films for THz modulation, Jepsen et al. and Zhang et al. have been identified in persistent studies. In studies conducted by Jepsen and his team, it was stated that VO₂ was used on a dielectric substrate for THz applications. The research findings indicated that VO₂ on a dielectric substrate transmits THz radiation below Tc, thereby exhibiting low conductivity and insulating properties. However, it has been demonstrated that above T_c, it reflects THz radiation, thereby acquiring metallic properties (Jepsen et al., 2006). Jepsen and his team employed THz time-resolved spectroscopy to examine the phase and amplitude of the broadband THz signal emitted by the VO₂ thin film on the dielectric substrate. In order to prevent the absorption of water vapour, the THz beam was maintained in a dry nitrogen environment. Furthermore, the entire assembly and sample were isolated from environmental influences by a thick layer in order to control the sample temperature. The optical properties of the VO2 thin film were examined between 25 °C and 100 °C. In order to ascertain the effect of temperature on the THz signal, the THz signal passing through the dielectric substrate alone was compared with the THz signal passing through the thin film at different temperatures. It has been demonstrated that at elevated temperatures, the film assumes a metallic state, resulting in a portion of the beam being reflected, thereby reducing the amplitude. Figure 1.2 compares the transmission of THz signals through the substrate at temperatures of 60 °C and 80 °C with the transmission through the substrate and the VO₂ film (Jepsen et al., 2006). Zhang and his team conducted a study utilizing the variable VO_2 in a switching mode. In the research, the authors examined VO_2 -embedded hybrid metamaterials (MM) containing cells consisting of two metals combined with VO_2 parts (Zhang et al., 2019). Experimentally, the modeswitching phenomenon in this metamaterial has been studied using conventional timeresolved spectroscopy (THz TDS) after thermal, electrical, and optical excitations. Comparative analysis of the responses to different stimuli indicates that thermal and electrical stimuli exhibit similar phase transition characteristics, whereas optical stimuli elicit a distinct response (Zhang et al., 2019).



Figure 1.2. Time-resolved Terahertz spectroscopy experimental setup (Jepsen et al., 2006)

1.5 Thesis' Motivation

The aim of this project is to determine the parameters required for the production of pure VO_2 thin films for various applications and to evaluate the suitability of the produced thin films for use by calculating the MIT ratios. In this context, the growth of VO_2 thin films has been carried out using changes in different parameters such as growth temperature, oxygen ratio, and coating time. In order to evaluate the quality of VO_2 thin films, their structural, electrical, and optical properties were examined. For the purpose of structural analysis, scanning electron microscopy (SEM) was used to characterize the surface morphology, Raman spectroscopy was used to characterize the vibration modes, and X-ray diffraction (XRD) was used to characterize the crystal structure. X-ray photoelectron spectroscopy (XPS) was used to detect the extent of surface contamination. Electrical characterization of produced VO₂ thin films plays a crucial role in determining MIT ratios. In this process, the current was applied from a current source with a probe placed between the two ends of the film, and then the voltage drop between the two probes was measured. The measured data were then used to calculate and evaluate the electrical resistance of the films in accordance with Ohm's Law. Furthermore, the measurements made it possible to determine the MIT of VO₂ at the Tc of approximately 68 °C. The analysis results enabled the determination of the most suitable parameters for subsequent growth processes by comparing the metal-insulator transition rates of VO₂ thin films produced with different growth parameters. These optimal parameters were determined by optimizing key variables such as growth temperature, oxygen-argon ratio, growth pressure, and voltage to ensure that the films exhibited the desired electrical and structural properties. Therefore, the motivation of this research is to produce VO₂ thin films that can be used in various applications.

CHAPTER 2

EXPERIMENTAL DETAILS

Chapter experimental details outline the techniques employed for the serial deposition and characterization of vanadium dioxide (VO_2) thin films on c-cut sapphire $[Al_2O_3 (0001)]$ by DC magnetron technique. To assess the quality of the films, a range of techniques were employed to examine their structural, electrical, and optical properties. Among these techniques, scanning electron microscopy (SEM) was used to analyze the surface morphology, Raman spectroscopy to investigate vibration modes, X-ray diffraction (XRD) to characterize the crystal structure, and X-ray photoelectron spectroscopy (XPS) to determine surface contamination. Electrical characterization techniques were also employed to determine the metal-insulator transition ratio.

2.1 Growth Process

The objective of this chapter is to provide an overview of the production methods for vanadium dioxide (VO_2) films. Magnetic sputtering, a type of physical vapor deposition (PVD), can be categorized into two types: Direct current (DC), and radio frequency (RF). In DC magnetic sputtering, the cathode (target) must be a negative conductor. In contrast, in RF magnetic sputtering, the target can be either a conductor or an insulator. The utilization of magnets in magnetic sputtering serves to confine secondary electrons emitted from the target, thereby enhancing excitation and ionization while reducing contamination on the substrate. Figures 2.1 and 2.2 provide a schematic representation of the processes occurring during the growth of vanadium dioxide within the chamber. The laboratory-built DC system was employed to facilitate the growth of vanadium dioxide, as illustrated in Figure 2.3. Given the complex nature of the chemical reaction between vanadium and oxygen, identifying a reliable method for growing vanadium dioxide is challenging. A multitude of variables affect the outcome of the thin film, necessitating the implementation of an augmentation procedure to ensure optimal growth conditions. It is common for different phases of vanadium oxides to coexist in a single sample, which highlights the significance of this procedure and its effective implementation.



Figure 2.1. Schematic image of magnetron sputtering (Yuce, 2015).

The growth of a uniform thin film is influenced by a number of factors, the temperature of the substrate, the pressure applied during deposition, and the position of the sample on the sample holder. These factors can be affected by a number of external conditions. Given the complex nature of the chemical reaction between vanadium and oxygen, identifying a reliable method for growing vanadium dioxide is challenging. A multitude of variables affect the outcome of the thin film, necessitating the implementation of an augmentation procedure to ensure optimal growth conditions. It is common for different phases of vanadium oxides to coexist in a single sample, which highlights the significance of this procedure and its effective implementation. In order to facilitate the growth process in the initial stage, the substrates were subjected to a cleaning procedure involving the use of acetone, methanol, and propanol alcohol, with each of these substances being used for a period of 10 minutes at each stage. This cleaning process was conducted using an ultrasonic vibrating cleaner.

The sample is then placed on the holder in a specific optimized position. The optimized position is defined as 13 mm from the center of the sample holder.

Parameter	Value
Base pressure	2.8 x 10 ⁻⁶ Torr
Deposition temperature	~ 520 °C
Argon flow	136 sccm
Oxygen flow	2.49 sccm
Pre-sputtering time	10 minutes
Deposition time	50 minutes
Distance between substrate and target	65 mm
Deposition pressure	5.5 x 10 ⁻³ Torr
Pre-sputtering power	70 W
Deposition power	70 W
Distance between holder's center and substrate	13 mm

Table 2.1. Optimum growth parameters determined for VO₂ thin film deposition



Figure 2.2. Schematic image of growth VO₂ (Ata, 2020).

The target held 6.5 cm from the sample holder, was made of 99.99% pure vanadium for magnification. Sapphire (c - Al₂O₃ (0001)), a material with high thermal conductivity (27.21 Wm⁻¹K⁻¹ at 300 K), was used as the substrate. The advantage of using sapphire as a substrate is its wide availability and high transparency in the THz range (Zhu et al., 2012). The base pressure was 2.8×10^{-6} Torr, and the chamber pressure during the growth process was 5.5×10^{-3} Torr. A 10-minute pre-scattering is required to remove contamination from the surface of the vanadium target and to ensure homogeneous growth on the substrate. The pre-scattering was performed with 70 W power and 40 sccm Ar gas. During deposition, 70 W DC power was applied to the V-target. During sputtering and deposition, the substrate is spun at approximately 12 rpm to form a homogeneous film. To obtain the VO₂ phase, 2.49 sccm O₂ and 136 sccm Ar were sent during thin film deposition by the MKS gas controller, and the pOxgen ratio is 1.80% according to equation 2.1 (Alaboz, 2018). A proportional-integral (PID) controller was coupled to a Jtype thermal couple in order to regulate the temperature, thereby obtaining the crystalline film by slowly heating the substrate to 520 °C and slowly cooling it after growth. In this process, four lamps with a power of 24 V and 250 W were used for heating. The heating lamps are placed 1 cm from the top of the substrate holder as shown in Figures 2.3 and 2.4.

$$\rho_{0_2} = [O_2(sccm)]/[O_2(sccm) + Ar(sccm)]$$
 (2.1)



Figure 2.3. Schematic image of DC magnetron sputtering (Aileen, 2023)



Figure 2.4. Original image of DC magnetron sputtering

2.2 Electrical Characterization

Vanadium dioxide (VO₂) thin films' resistance produced in our laboratory in the Physics Department of IZTECH was measured using a probe station device (Janis Microprobe Station) as shown in Figure 2.5. Vanadium dioxide exhibits thermal hysteresis during heating to 100 °C and cooling to room temperature. Its behavior during cooling tends to be more stable than during heating. Using a Labview program written by us and a Keithley 2100 digital multimeter, the temperature-dependent resistance change was measured with a probe station. Applied silver paste to the edges of the thin films to get electrical contact between the VO₂ thin film and the probe rod. In the electrical measurement, thermal gel was employed to facilitate the requisite thermal transition during the heating process for temperature-resistance dependence. Under vacuum conditions, the sample temperature was gradually increased to 100 °C until the metal-insulator transition of the film was observed at about 68 °C, as shown in the graph. Measurements were taken during cooling as the temperature gradually decreased after the heater was turned off. This shows that the VO₂ film was grown quite successfully and in a single phase when the resistivity of the film changed by a factor of 10⁴.



Figure 2.5. Image of Janis microprobe station.

2.2.1 Resistance Measurement by Two Probe Methods

The Two Probe Method is a technique employed to assess the electrical resistivity of specific materials. The objective of this method is to gain insight into the electrical properties of the material and to evaluate its suitability for diverse applications. Two metal probes are positioned between the two ends of the material, with the intention of detecting the voltage required to measure the electrical resistance of the material. Initially, direct current is applied to one end of the material from a current source, whereby the current is transmitted through the material. The voltage drop between the two probes is then measured, and this voltage is subsequently employed to calculate the resistance of the material in question (Khalid, 2022). Subsequently, the voltage drop between the two probes is quantified, and this voltage is employed to determine the resistance of the material. Measurements of VO₂ thin films were conducted using silver paste, with many samples measuring 1 cm x 1 cm.

2.3. SEM And EDX Characterization

A scanning electron microscope (SEM) generates high-resolution images through the use of electrons. These electrons are typically emitted from a tungsten or lanthanum hexaboride filament source. Once emitted, the electron beam is focused into a fine spot by a series of electromagnetic lenses. This finely focused electron beam is then directed onto the sample surface, resulting in the bombardment of the sample. The interaction between the sample surface and the electron beam results in the emission of a variety of signals, predominantly secondary electrons, which are subsequently collected to form images. The scattered electrons are detected by a detector positioned at an additional electrical potential. This detector converts the electron signal into an image that provides detailed information about the surface morphology of the sample. The result is a threedimensional representation of the surface topography, providing insight into the structural and compositional properties of the sample at the micro- and nanoscale. The surface composition and morphology of VO₂ thin films were examined using the Oxford X-act Energy Dispersive X-ray Spectroscopy (EDX), and FEI-QuantaFEG 250 Scanning Electron Microscope (SEM) at TAM of IZTECH. The surface topography was imaged using a Circular Backscatter Detector (CBS) and an accelerating voltage of 7 kV at 10,000X magnification in a high vacuum with a spot size of 4.0. Energy-dispersive X-ray spectroscopy (EDS) analysis was conducted using a 500X magnification, backscattered electron detector, 20 kV resolution, and 7.0 spot size in a high vacuum.

2.4 X-Ray Diffraction

X-ray powder diffraction (XRD) is utilized to determine the structure of crystalline materials due to their distinct atomic spacing. This method operates on the principles of Bragg's Law, which establishes a relationship between the wavelength and angle of the incoming X-ray beam and the spacing between the crystal's atomic layers. In equation 2.2, d represents the distance between these layers, θ is the angle of the X-ray incidence, n is an integer representing the order of diffraction, and λ is the wavelength of the X-ray. Given that the X-ray wavelength closely matches the spacing between atoms, this method is ideal for ascertaining the crystalline structure's orientation. The X-ray diffraction analysis was conducted using a Phillips X'Pert Pro diffractometer in Bragg-

Brentano focusing geometry with Cu K α radiation ($\lambda = 1.5456$ Å). XRD scans covered an angular range from $2\theta = 10^{\circ}$ - 90° with a step size of 0.001° for each sample.



$$n\lambda = 2dsin\theta \tag{2.2}$$

Figure 2.6. X-ray diffraction model (Gottimukkala, 2005)

2.5 Raman Spectroscopy

During the analyzes of light scattered by a molecule or crystal, the predominant portion of photons undergo elastic scattering, wherein the wavelength of the scattered light remains unchanged from that of the incident light. This type of scattering is referred to as Rayleigh scattering. Conversely, When the wavelength of the scattered light differs from that of the incident light, the scattering is considered inelastic and is referred to as the Raman effect. This type of scattering involves a slight energy shift in the scattered light caused by the interaction between the laser light and molecular vibrations or phonons, as explained by Turrell and Corset in 1996. The shift in energy between the incident and scattered light is described by Equation 2.4, where \mathcal{V} represents the Raman shift (cm⁻¹) and λ denotes the wavelengths of the incident and scattered light in Raman spectroscopy.

$$\mathcal{V} = \frac{1}{\lambda \text{ind.}} - \frac{1}{\lambda \text{scat}}$$
(2.4)

Confocal Raman microscopes have been employed to conduct Raman measurements at a range of excitation wavelengths, encompassing the visible and infrared regions. The microscopes include the inVia Raman system from Renishaw, the WiTec alpha300 confocal Raman system, and the RXN system from Kaiser Optical Systems, Inc., Arbor, MI, USA. In particular, Raman spectra were generated using a WiTec alpha300 confocal microscope, with excitation provided by a 532 nm Nd-YAG green laser.

2.6 X-Ray Photoelectron Spectroscopy

X-ray photoelectron spectroscopy (XPS) is a widely used method for analyzing the surface chemistry of materials. This method, which is frequently employed in fields such as chemistry, materials science, physics, and engineering, is effective in determining the chemical compositions and oxidation states of the elements on the surface of the material. XPS commences with the irradiation of the sample with monochromatic X-rays. The X-rays possess sufficient energy to excite electrons at the nuclear level of atoms within the material. X-ray photons interact with electrons at the core level of atoms, resulting in the release of these electrons from the surface. These released electrons are referred to as photoelectrons. The kinetic energies of photoelectrons are quantified by an electron spectrometer, which is then employed to ascertain the binding energies of electrons within the atom. The binding energy is determined using equation 2.5, where E_B denotes the binding energy, hv is the energy of the X-ray photons, E_K represents the kinetic energy of the photoelectron, and ϕ is the spectrometer's work function (Paterson and Swaffield, 1994).

$$E_B = hv - E_K - \phi \tag{2.5}$$

The resulting spectrum displays peaks that correspond to the binding energies of different elements and their chemical states. The intensity of these peaks is proportional to the amount of the corresponding element on the surface. XPS typically analyses the upper 1-10 nanometres of the material and is a powerful tool for determining the types and amounts of elements, chemical bonds, and electronic structures on the surface. This

technique enables the quantification of the elemental composition of the surface by measuring the area under the peaks in the XPS spectrum and applying sensitivity factors. XPS measurements of VO₂ films, performed using a non-monochromatic Mg Ka radiation source (hv = 1254 eV) with a power of 150 W and a separation angle of 45°, were carried out using SPECS Phoibos 150 3D-DLD. Valence region spectra of V2p, O1s, and C1s were obtained by setting the analyzer's transition energy to 30 eV, step size to 0.05 eV, dwell time to 2 s, and spot size to 2 mm.

CHAPTER 3

RESULTS AND DISCUSSION

This chapter presents a discussion of the characterization techniques employed to ascertain the requisite parameters for the mass production of vanadium dioxide (VO₂) thin films on c-cut sapphire [Al₂O₃ (0001)] via the DC magnetron technique. During the mass production process, the structural, and electrical properties of the thin films were examined in order to evaluate their quality. These methods, which are critical for the deposition and evaluation of thin films, enable the comparison of films according to their MIT rates. In the electrical characterization section, a number of techniques were employed to ascertain the metal-insulator transition ratio of thin films. In this context, a number of analytical techniques have been employed, including scanning electron microscopy (SEM) to analyze surface morphology, Raman spectroscopy to investigate vibration modes, X-ray diffraction (XRD) to characterize the crystal structure, and X-ray photoelectron spectroscopy (XPS) to determine surface contamination. The electrical characterization of thin films plays a pivotal role in determining the metal-insulator transition ratio. In this process, a probe is placed between the two ends of the film, with a current source applied to one end of the material. The voltage drop between the two probes is then measured. The electrical resistance of the film is calculated based on Ohm's Law, using the measured voltage and current values. These characterization techniques permit the detailed analyzes of the structural, electrical, and optical properties of VO₂ thin films grown on c-cut sapphire. This analyzes provides the necessary parameters for the mass production and quality assessment of the films.

3.1 SEM-EDX

The sample was analyzed using SEM and EDX to investigate the surface morphology and stoichiometry. The surface topography was imaged using a Circular Backscatter Detector (CBS) and an accelerating voltage of 7 kV at 10,000X magnification in a high vacuum with a spot size of 4.0. Energy-dispersive X-ray spectroscopy (EDX) analyzes was conducted using a 500X magnification, backscattered electron detector, 20 kV resolution, and 7.0 spot size in a high vacuum.



Figure 3.1. Sem images

Because of the EDX analyzes technique during elemental analyzes from sapphire (Al₂O₃) also gains oxygen and aluminum (Figure 3.2). That's why, the elemental rate seems different. Other types of vanadium oxide phases cannot be detected from the atomic weight percentage of EDX analyzes (Martinez et al., 2022).



Figure 3.2. EDX analyzes

3.2 X-Ray Diffraction (XRD) Analyzes

X-ray diffraction analysis has a critical role in investigating crystal structures of thin films. VO₂ materials have unique properties in that crystal structure changes from monoclinic at low temperatures to rutile phase at high temperatures. And in this thesis, investigated VO₂ thin films' monoclinic phase to obtain pure VO₂ phase except for other vanadium oxide phases (V₂O₃ and V₂O₅, etc.). The analysis was made by Phillips X'Pert Pro X-Ray diffractometer in Bragg-Brentano focusing geometry using Cu Ka radiation $(\lambda = 1.5456 \text{ Å})$. XRD patterns were collected in the $2\theta = 10^{\circ}$ - 90° range for all samples with a step size of 0.001°. XRD cards (00-010-0173 Al₂O₃ and 00-019-1398 VO₂) were used to obtain Miller indices. As shown in Figure 3.3 there are main peaks in 37°, 40°, and match with literature the spectrum 37°, 39.5° which are related to planes (200), (020) of VO₂ (Kim et al. 2014) and 41.5° Al₂O₃. This thesis couldn't make thickness measurements. However, to compare VO₂ thin film's thickness dependence with deposition time. Selected two thin films that have the same growth parameters except time. One of them has 50 minutes of deposition time and the other 45 minutes. If compared Figure 3.3 shows the XRD analysis of VO₂ thin film with 50 minutes and Figure 3.4 shows the XRD analysis of VO₂ thin film with 45 minutes. The sapphire substrate has a bigger intensity in Figure 3.4, which means the thin film's thickness is smaller than others.



Figure 3.3. XRD analyses in the range 36-46 for VO₂ thin film had a deposition time of 50 minutes.



Figure 3.4. XRD analyses in the range 36-46 for VO₂ thin film had a deposition time of 45 minutes.

3.3 Raman Spectroscopy Analyzes

The analysis of grown thin films by Raman spectroscopy represents a pivotal stage in the evaluation of the vibration modes and structural properties of the films in question. Raman spectroscopy is a highly effective technique employed to ascertain the crystal structure of the material and the state of the chemical bonds within it, thereby furnishing crucial insights into the phases that emerge during the deposition of VO₂ thin films. In this thesis, two thin films were grown under identical conditions and the optimal growth parameters were identified. It was demonstrated that VO₂ thin films can be successfully produced. As depicted in Figure 3.5, peaks at 313 cm⁻¹, 392 cm⁻¹, 502 cm⁻¹ and 617 cm⁻¹ were obtained. Three graphs exhibited peaks consistent with the literature, where 260, 311, 338, 389, 441, and 494 cm⁻¹ are known to correspond to VO₂ vibration modes at room temperature (Vikhnin et al., 1995). The peak observed at 617 cm⁻¹ signifies the main peak of the VO₂ phase and is associated with the V-V vibration mode (Piccirello et al., 2009).



Figure 3.5. Two thin films grow with the same parameters that 520 °C, 5.5×10^{-3} Torr, 2.49 sccm O₂ flow.

3.4 XPS Analyzes

The sample numbered BA-VO₂-65, obtained as a result of magnetic sputtering, was taken from the vacuum chamber where the growth took place and placed in the XPS device, ensuring that there was no contamination on it, thus preserving the purity of the thin films when they were grown, and elemental analyzes were carried out on the surfaces of the thin films. During the analysis using x-rays from magnesium K α radiation, the thin film was held on with copper holders. As a result of the measurements, no other contaminating material was found in the films as shown in Figure 3.6, except for the Carbon element that could be observed on the surface due to the sensitivity of XPS measurements. XPS measurements were carried out in an area of 2 mm diameter at a depth of 10 nm. Due to the very sensitivity of the measurements on the surface and the easy contamination of carbon, the formation of a uniform peak when removing ~284 eV on the sample surface shows that it does not interact with VO₂ (Figure 3.7). As can be seen in the high-resolution measurements in Figure 3.8, V 2p peaks appear in the range of ~532 eV O 1s and ~525, 510 eV.



Figure 3.6. Survey of BA-VO₂-65 thin film that growth parameters are 550 oC, 50minutes, 5.5 x 10-3 Torr



Figure 3.7. High resolution of Carbon region of BA-VO₂-65 thin film.



Figure 3.8. High resolution of vanadium oxide region

3.5 Electrical Characterization

Electrical characterization of the VO₂ thin films produced after the enlargement and monitoring of different copies such as temperature, oxygen rate, and deposition time were performed. As a result of this investigation, a parameter set was determined for the metal-insulator transition ratios (MIT ratios) of the films obtained. These calculations determine which parameter policies and records to determine the best results. In particular, different temperatures, oxygen ratios, and coating conditions have significant effects on the phase transition temperature and conductivity properties of VO₂. During the electrical characterization process, the voltage between the two probes was stopped by applying current from a current source with the probes separated from each other at the two ends of the films. These measurements were recorded and evaluated according to Ohm's Law to program the electrical resistance of the thin films. The obtained data were related to the MIT of VO₂ films at critical points at approximately 68 °C. As a result of the analyses, the ideal effect for subsequent growth processes was determined by comparing the metal-insulator transition rates of the produced VO₂ thin films. The VO₂ growth process has several critical points; growth temperature, oxygen-argon ratio, growth pressure, power, etc. VO_2 has different phases example as V_2O_3 and V_2O_5 , other phases have different critical temperatures for metal-insulator transition. The determination of the MIT ratio is contingent upon the implementation of electrical characterization as well as determining the critical temperature around 68 °C.

Sample name		Sample	Growth	
Sumpre nume	$O_2(scem)$	resistance	Temperature	MIT rate
	02(00011)	(Kilo ohm)	(°C)	
BA-VO ₂ -6	2.26	6.10	550	2.36
BA-VO ₂ -14	2.48	20	547	2.98
BA-VO ₂ -15	2.50	27	552	3.38
BA-VO ₂ -16	2.52	370	550	2.64
BA-VO ₂ -21	2.49	80	560	3.89
BA-VO ₂ -22	2.47	28	560	3.24
BA-VO ₂ -24	2.49	30	551	3.35
BA-VO ₂ -25	2.49	17	550	3.26
BA-VO ₂ -26	2.49	20	550	3.24
BA-VO ₂ -28	2.49	40	550	3.31
BA-VO ₂ -29	2.49	18	550	3.12
BA-VO ₂ -31	2.49	24	550	3.33
BA-VO ₂ -37	2.49	6	550	2.77
BA-VO ₂ -38	2.49	5.6	550	2.70
BA-VO ₂ -39	2.49	153	550	2.66
BA-VO ₂ -40	2.49	145	550	1.98
BA-VO ₂ -41	2.49	77	550	3.55
BA-VO ₂ -42	2.49	37	550	2.98
BA-VO ₂ -43	2.49	36	550	2.95
BA-VO ₂ -45	2.49	144	530	2.65
BA-VO ₂ -46	2.49	53	540	3.57
BA-VO ₂ -47	2.49	32	530	3.58
BA-VO ₂ -48	2.49	63	520	3.60
BA-VO ₂ -49	2.49	36	510	3.31
BA-VO ₂ -50	2.49	36	540	3.32
BA-VO ₂ -52	2.49	14	515	3.10
BA-VO ₂ -53	2.49	25	525	3.20
BA-VO ₂ -55	2.55	28	520	3.25
BA-VO ₂ -56	2.49	50	520	3.47
BA-VO ₂ -57	2.49	20	520	3.00
BA-VO ₂ -58	2.49	24	520	2.98
BA-VO ₂ -59	2.49	21	520	3.07
BA-VO ₂ -60	2.49	72	520	3.18
BA-VO ₂ -61	2.49	68	520	3.36
BA-VO ₂ -62	2.49	66	527	3.42
BA-VO ₂ -63	2.49	69	520	3.52
BA-VO ₂ -64	2.49	75	520	3.19
BA-VO ₂ -65	2.49	32	520	2.69
BA-VO ₂ -66	2.49	101	520	2.95

Table 3.1. VO₂ thin films' growth parameters and MIT rates

In this thesis, compare the MIT ratio for different thin films that grow with different parameters. Shown Figure 3.9; BA-VO₂-21 has $10^{3.89}$, BA-VO₂-24 has $10^{3.35}$, BA-VO₂-25 has $10^{3.26}$, BA-VO₂-28 has $10^{3.31}$ and, BA-VO₂-29 has $10^{3.12}$ MIT change rate. When compared with 50, 55, and 45-minutes growth time. Samples that grow 50 minutes have a bigger MIT transition than other samples. From Figure 3.9, we can mention that the 50-minute growth process is the best parameter. Thin film thickness is

lower for less growth minutes like 45 minutes. And, when increased growth time can be caused by other phases of vanadium dioxide.



Figure 3.9 MIT ratios of VO₂ thin films that grow with different times.

Shown Figure 3.10; BA-VO₂-46 has $10^{3.57}$, BA-VO₂-47 has $10^{3.58}$, BA-VO₂-48 has $10^{3.60}$, and BA-VO₂-49 has a $10^{3.31}$ MIT ratio. A comparison of the thin films at different temperatures revealed that the temperature of 520 °C was superior to the others. Furthermore, the experimental setup allows for the temperature to be stabilized at 520 °C, which is a more comfortable process. Consequently, a significant number of depositions were conducted at 520 °C.



Figure 3.10 MIT ratios of VO₂ thin films that grow with different temperatures.

Shown in Figure 3.11; BA-VO₂-6 has $10^{2.38}$, BA-VO₂-14 has $10^{2.98}$, BA-VO₂-21 has $10^{3.89}$, BA-VO₂-22 has $10^{3.24}$, BA-VO₂-24 has $10^{3.35}$ MIT rate. When we compare thin films' MIT rate with different oxygen-argon ratios, 2.49 sccm O₂ with 1.8% ratio better sccm parameter to growth VO₂ as shown in Figure 3.11. Vanadium dioxide (VO₂) thin films were produced by depositing with using a vanadium target and oxygen gas. Consequently, the oxygen ratio assumes a pivotal role in the VO₂ phase, serving to preclude the formation of other phases.



Figure 3.11 MIT ratios of VO₂ thin films that grow with different oxygen flow ratios, and 2.49 sccm growth two times because of the ensure that this sccm ratio is better than the others.

Although the metal-insulator transition is a significant feature of VO₂, it must be possible to reverse this transition. As a consequence of the irreversible phase change, the tunability feature of metallic VO₂ is lost, which gains permanent metallic properties. Consequently, the transition from insulator to metal, which occurs initially when heated to 25-100 °C, should also be reversible during the cooling process. In order to ascertain this feature, hysteresis plots were carried out. Figure 3.12 illustrates the hysteresis of VO₂ thin film that has a $10^{3.89}$ MIT rate. Figure 3.13 illustrates that even when the MIT value is low, the reversible feature of $10^{2.65}$ is maintained despite its initial tendency towards metallicity.



Figure 3.12. Hysteris of the thin film that has a large MIT is $10^{3.89}$



Figure 3.13. Hysteris of the thin film that has a small MIT is $10^{2.65}$

CHAPTER 4

CONCLUSION

Vanadium dioxide (VO₂) is a material that is known to exhibit a metal-insulator transition (MIT) property. The material undergoes a phase transition from the insulating to the metallic phase at approximately 68 °C (Yuce, 2015). During this transition, the material's electrical conductivity and optical properties undergo a significant change, rendering VO₂ an optimal choice for a multitude of electronic and optical applications, including sensors, switches, and memory devices (Pergament et.al, 2013). The phase transition of VO₂ occurs when the material transforms from a monoclinic structure at low temperatures to a rutile structure above ~68 °C temperature. In the metallic phase, VO₂ exhibits high electrical conductivity, whereas in the insulating phase, it displays low conductivity. This characteristic renders VO₂ an appropriate material for use in advanced technological applications, including energy-saving smart windows, terahertz (THz) wave modulators, and Mott transistors (Noori, 2023). The objective of this thesis is to ascertain the requisite parameters for the growth of pure VO₂ thin films and to assess the suitability of the produced thin films for use by calculating the MIT ratio. The metalinsulator transition (MIT) is a distinctive feature of VO₂ thin films, rendering them suitable for a variety of devices. In order to ascertain this transition rate, the temperature dependence of the resistance was examined. The determination of the MIT transition was contingent upon the implementation of electrical measurements. By applying a current from a current source with the probe placed between the two ends of the thin films, the voltage drop between the two probes was measured. The measured data were then used to calculate and evaluate the electrical resistance of the films in accordance with Ohm's Law. This process permitted the determination of the MIT of VO₂ at the Tc ~68 °C. The results of these analyses enabled the determination of the optimal parameters for subsequent growth processes by comparing the MIT ratios of VO₂ thin films produced with different growth parameters. It has been demonstrated that thin films produced with optimal parameters exhibit high electrical conductivity and provide the desired performance properties. These findings lend support to the proposition that VO₂ thin films can be efficiently deployed in advanced electronic and optical applications. The results

obtained using structural characterization techniques are as follows: In the Raman spectroscopy analyses conducted at a wavelength of 532 nm, peaks were observed at 313 cm⁻¹, 392 cm⁻¹, 502 cm⁻¹, and 617 cm⁻¹ for two samples. These peaks correspond to the characteristic vibration modes of VO₂, thereby confirming the structural integrity of the films. X-ray diffraction analyses revealed the presence of peaks at 37°, and 39.5°, indicating that the crystal structure of the films is preserved and is consistent with the values reported in the literature. Furthermore, X-ray photoelectron spectroscopy was conducted to ascertain the presence of surface contamination. The analyses revealed the presence of carbon, but no other surface contamination was detected. These findings corroborate the assertion that the films are free from surface contamination and exhibit overall quality. SEM and EDX analyses were employed to assess the surface morphology and elemental stoichiometry, respectively. The results demonstrated that the internal structure of the films remained intact, indicating that no factors had caused any damage. Consequently, the quality of the manufactured thin films was assessed, and it was established that they were appropriate for utilization in a multitude of electronic and optical applications.

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