

**PHOTORESPONSIVITY ENHANCEMENT OF PLASMA
ASSISTED CdTe THIN FILMS FOR PHOTOVOLTAIC
APPLICATIONS**

BY

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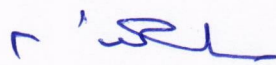
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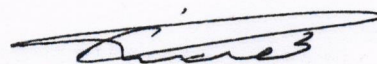


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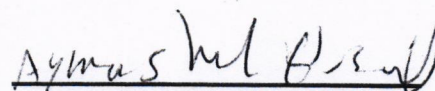
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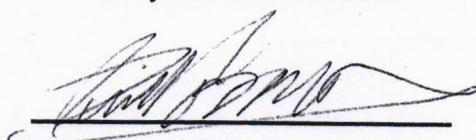
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DEDICATION



*"Whoever follows a path in which he seeks
knowledge, Allah has facilitated for him a path
to Paradise"*

PROPHET MUHAMMAD (PEACE BE UPON HIM)

*Dedicated to My Parents, Husband, Daughter,
Brother, and respected teachers.*

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ABSTRACT

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Photovoltaic (PV) technology presents the most effective answer to the challenges of energy shortages and the need for cleaner energy sources. Thin film semiconductors hold significant promise for the development of optoelectronic and photovoltaic devices. Among the II-VI binary semiconducting compounds, cadmium telluride is recognized as one of the most promising photovoltaic materials, especially for application in thin-film solar cells. It has achieved notable conversion efficiencies of over 22% and is regarded as the second-lowest-cost material after silicon in the photovoltaic market. Post-deposition plasma treatment of the films has a significant effect on their properties. In plasma etching, a gas such as O₂, Ar, N₂, SF₆, CF₄, C₂H₅OH, or their mixture is used as an etchant gas. In this work, CdTe thin films were prepared by thermal evaporation and subsequently subjected to Ar plasma treatment for one hour with an argon flow rate of 20, 30, and 40 sccm at different plasma powers. The structural properties were investigated using X-ray diffraction (XRD). Moreover, the morphological properties were studied using an Atomic Force Microscope (AFM). The optical properties were studied by obtaining the transmittance spectrum and calculating the band gap values. Then, the current-voltage curves were obtained using a longitudinal configuration by sweeping the voltage from 0 to

10 V and measuring the corresponding current. All measurements were conducted in the dark and with a 75 W xenon lamp illumination.

ABSTRACT (ARABIC)

ملخص الرسالة

الاسم: الاء الوحيشي

عنوان الرسالة : اقامة تعديلات بواسطة البلازما علي اسطح أغشية الكاديوم تيليريد الرقيقة المحضرة بواسطة التبخير الحراري من أجل تعزيز استجابتها الكهروضوئية.

مجال التخصص: فيزياء

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تقدم تقنية الطاقة الكهروضوئية الإجابة الأكثر فعالية على تحديات نقص الطاقة والحاجة إلى مصادر طاقة أنظف. تحمل أشباه الموصلات ذات الأغشية الرقيقة املاً كبيراً لتطوير أشباه الموصلات الكهروضوئية والأجهزة الكهروضوئية. من بين مركبات اشباه الموصلات الثنائية يبرز مركب الكاديوم تيليريد كواحد من أكثر مواد الطاقة الكهروضوئية الواعدة وخاصة للاستخدام في الخلايا الشمسية ذات الاغشية رقيقه. لقد حقق كفاءات تحويل ملحوظه تزيد عن 22% ويعتبر ثاني أقل مادة تكلفة بعد السليكون في سوق الطاقة الكهروضوئية. كما أن معالجة المواد بالبلازما بعد الترسيب للأغشية الرقيقة لها تأثير كبير علي خصائصها. في عملية المعالجة بالبلازما يتم استخدام غاز مثل الاكسجين او الارجون او النيتروجين او خليط منها كغاز معالج. في هذا العمل تم تحضير أغشية رقيقة من الكاديوم تيليريد عن طريق التبخير الحراري ثم تعرضت للمعالجة ببلازما الأرجون لمدة ساعة بمعدل تدفق للأرجون 20 و 30 و 40 سم مكعب في الدقيقة عند قوى بلازما مختلفة.تم دراسة الخصائص البنيوية باستخدام تقنية حيود الاشعة السينية .علاوة علي ذلك تمت دراسة الخصائص المورفولوجية باستخدام مجهر القوة الذرية. بالاضافة الي ذلك تمت دراسة الخصائص البصرية من خلال الحصول علي طيف النفاذية وحساب قيم فجوة النطاق. بعد ذلك تم الحصول علي منحنيات للتيار الكهربائي مقابل الجهد

الكهربي باستخدام التكوين الطولي وذلك عن طريق تغيير الجهد من 0 الي 10 فولت وحساب قيمة التيار الكهربائي المقابل. أجريت جميع القياسات في الظلام وفي وجود الضوء مقدمه من مصباح زينون بقوة 75 وات.

CHAPTER 1

INTRODUCTION

1.1 PHOTOVOLTAIC MATERIALS

Photovoltaics (PV), which is concerned with the transformation of solar energy into electricity, provides a viable and sustainable solution to the growing global energy demand. Becquerel was the first to observe the phenomenon of photovoltaics [1]. The phenomenon is typically characterized by the generation of an electric voltage in a solid or liquid when illuminated by light. While photovoltaic devices generate electricity upon exposure to sunlight, various additional components are necessary to effectively conduct, regulate, convert, distribute, and store the energy generated by the device. Based on the system's functional and operational requirements, essential components may encompass a DC-AC power inverter, battery bank, system and battery controller, auxiliary energy sources, and occasionally the designated electrical load (appliances). Furthermore, a variety of balance of system (BOS) components, encompassing wiring, overcurrent protection, surge protection, disconnect devices, and additional power processing apparatus. Figure 1.1 illustrates a fundamental diagram of a photovoltaic system and the interconnection of its components [4].

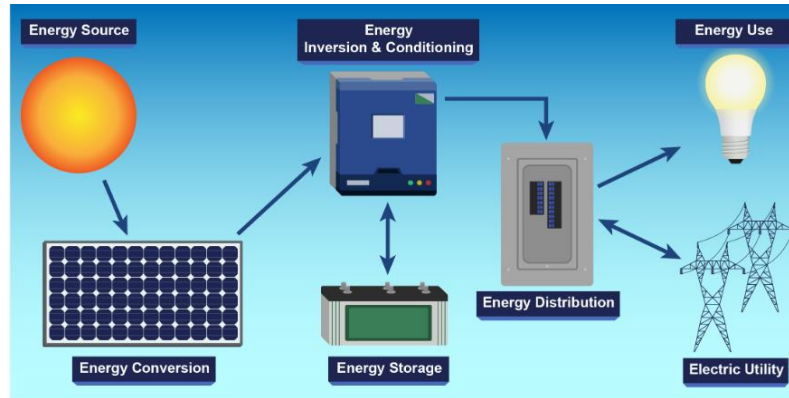


Figure 1.1 A basic diagram of a photovoltaic system and the relationship of individual components.

As shown, once the energy is absorbed, the energy is converted to electricity depending on the nature of the photovoltaic effect. A photovoltaic cell is fundamentally a large-area p–n semiconductor junction that captures photon energy to generate electrical energy. At the semiconductor level, the p–n junction establishes a depletion region characterized by an electric field oriented in a certain direction. When a photon with adequate energy strikes the material in the depletion region, the photon's energy excites a valence electron into the conduction band, resulting in a vacancy in the valence band. The electric field in the depletion region causes electrons and holes to move in opposing directions, resulting in a net current. Figure 1.2 illustrates the process by which a photon generates an electron-hole pair [3]. The current produced by the voltage generated at the semiconductor junction enables the photovoltaic cell to generate direct current power.

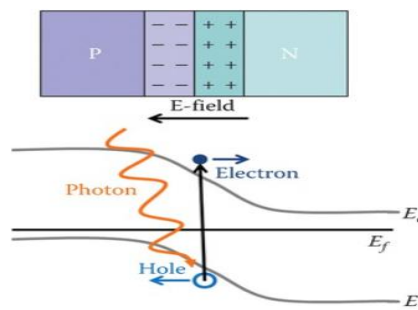


Figure 1.2 The process of a photon generating an electron-hole pair in a PV cell.

For semiconductors, which are one class of materials used to produce solar cells, there is a characteristic term called band gap. A band gap, also referred to as a bandgap or energy gap, is an energy range in a solid devoid of electronic states [4,5]. In the electronic band structure of a semiconductor, the band gap denotes the energy difference between the valence band's top point and the conduction band's lowest point. It is the energy necessary to elevate an electron from the valence band to the conduction band. As shown in the solar spectrum Figure 1.3, photons possessing energies lower than the band gap are not absorbed [4,5]. Conversely, photons with energies exceeding the band gap are not wholly converted into electrical energy due to the thermalization of charge carriers. The challenges associated with practical solar cells underscore the need for further research and innovation [3-6]. Considering these two factors, roughly 45% of the incident spectrum-integrated solar power is accessible for semiconductors with a band gap between 1.1 and 1.4 eV. This denotes the peak power produced when the cell functions at a voltage corresponding to the band gap energy and a current that completely harnesses all photons surpassing the band gap, in addition to the total accumulation of all generated carriers. In practical solar cells, not all incident light is absorbed by the active layers, and not all generated charge carriers are collected. Consequently, the short-circuit current (J_{sc}) is less than the maximum attainable value for a specific band gap (E_g). The attainable open-circuit voltage (V_{oc}) falls short of the Shockley-Queisser limit, which represents the maximum theoretical efficiency of a solar cell employing a single p-n junction to capture energy, with radiative recombination being the only loss mechanism in the solar cell [3.5]. It was first calculated by William Shockley and Hans-Joachim Queisser at Shockley Semiconductor in 1961, giving a maximum efficiency of 30% at 1.1 eV. The reason for that is various factors,

including Auger recombination, band tail recombination, and recombination occurring at bulk, interface, and surface defects [4-6]. Additionally, resistance, contact losses, and various non-idealities contribute to a reduction in the fill factor (FF). The identified factors contribute to practical efficiencies that frequently fall significantly below the Shockley-Queisser limit associated with a specific band gap [3].

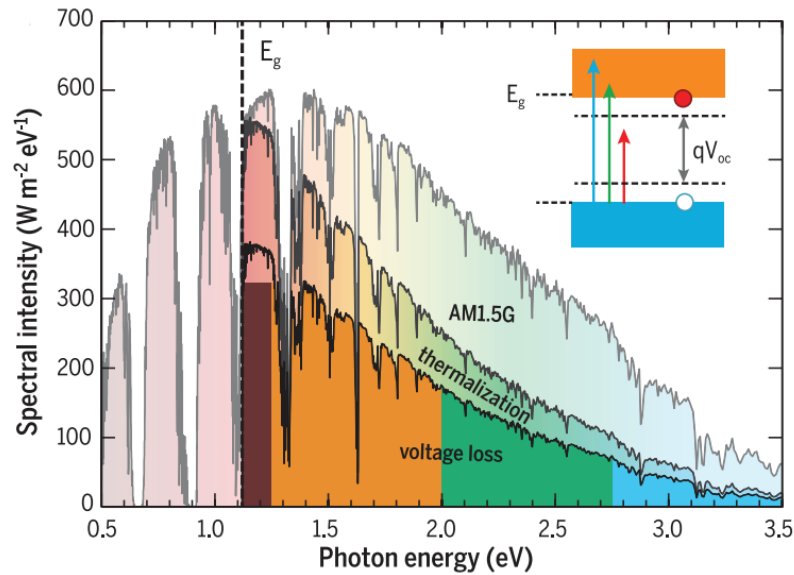


Figure 1.3 The solar spectrum.

1.2 CLASSES OF PV MATERIALS

Photovoltaic materials can be classified into three categories: (i) Ultrahigh-efficiency monocrystalline materials exhibit efficiencies exceeding 75% of the Shockley-Queisser limit for their respective band gaps, including silicon (both homojunction and heterojunction), gallium arsenide (GaAs), and gallium indium phosphide (GaInP); (ii) high-efficiency polycrystalline materials achieve 50 to 75% of the Shockley-Queisser limit,

such as silicon, copper indium gallium selenide/sulfide (CIGS), cadmium telluride (CdTe), methyl ammonium lead halide perovskite [$\text{CH}_3\text{NH}_3\text{Pb}(\text{I}, \text{Cl}, \text{Br})_3$], and indium phosphide (InP); and (iii) low-efficiency materials fall below 50% of the Shockley-Queisser limit, including micro or nanocrystalline and amorphous silicon (a-Si:H), copper zinc tin selenide/sulfide (CZTS), dye-sensitized titanium dioxide (dye / TiO_2), organic and polymer materials, and quantum dot materials (PbS QD) [3]. Figure 1.4 illustrates the optimal efficiency for each material [3].

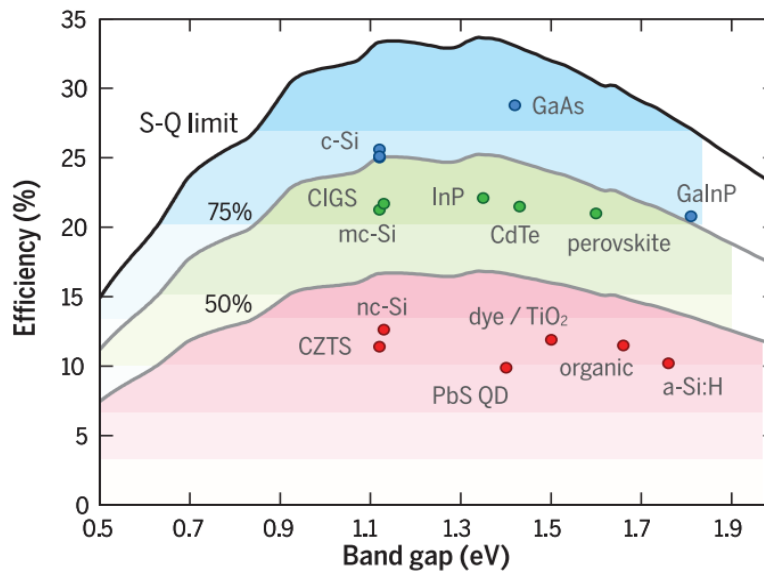


Figure 1.4 The optimal efficiency for solar energy materials.

However, the reality is that producing a perfect solar cell with high efficiency is not a simple task. It requires an optimal solar cell material with a direct band gap ranging from 1.1 to 1.7 eV, composed of affordable, non-toxic substances, and can be deposited by reproducible deposition methods appropriate for large-scale manufacturing to be stable for a long time [7]. Unfortunately, a material fulfilling all these requirements has not yet been

found. This underscores the urgency and importance of ongoing research in this field. Silicon, for instance, possesses an almost optimal band gap of approximately 1.12 eV and can achieve a high efficiency of 25.6%. However, its indirect band gap leads to a significantly low absorption coefficient that changes gradually near the band gap energy. Consequently, a wafer of relatively considerable thickness is necessary to absorb all photons with energies exceeding the band gap [8-11]. This, however, results in increased bulk (Auger) recombination, thereby diminishing V_{oc} . Additionally, it raises the cost of materials. Organic solar cells, on the other hand, facilitate cost-effective roll-to-roll manufacturing on flexible substrates and provide a diverse selection of materials suitable for applications requiring flexibility and color diversity. The maximum efficiency achieved for a single-junction organic solar cell is 11.5% [8,9]. The low dielectric constant of organic materials leads to tightly bound photogenerated electron-hole pairs, requiring specialized architectures like bulk heterojunctions for effective charge separation and extraction. The energy offsets require for the heterojunction to facilitate efficient exciton dissociation results in a voltage loss of approximately 0.3 V, leading to a reduction in efficiency by about 2% [10]. Moreover, the principal obstacles for organic solar cells are the high rate of nonradiative recombination and considerable static and dynamic disorder, leading to significant voltage loss ($V = 0.5V$)[3,10].

1.3 THIN FILM SOLAR CELLS

In discussions regarding solar technology, the predominant focus is often on crystalline silicon (c-Si) panels. Although this technology is the most prevalent, an alternative with a

promising future is thin-film solar technology. Thin-film solar technology has existed for decades and has demonstrated its efficacy by offering numerous versatile and distinctive applications unattainable by crystalline silicon solar cells. Thin-film solar panels employ a second-generation technology distinct from crystalline silicon (c-Si) modules, which are the predominant technology. Thin-film solar cells (TFSC) are produced by applying single or multiple layers of photovoltaic elements onto a substrate made of various materials, including glass, plastic, or metal. Also, thin-film solar panels require less semiconductor material in the manufacturing process than regular crystalline silicon modules since the thickness of a thin-film layer is in the range of a few micrometers, more than 20 times thinner than the thinnest silicon wafers [2,11,12]. Nonetheless, they function comparably under the photovoltaic effect. To have an idea about the importance of manufacturing thin film solar cells, Figure 1.5 illustrates the predicted thin film solar cell market size from 2022 to 2032 (USD billion) [13].

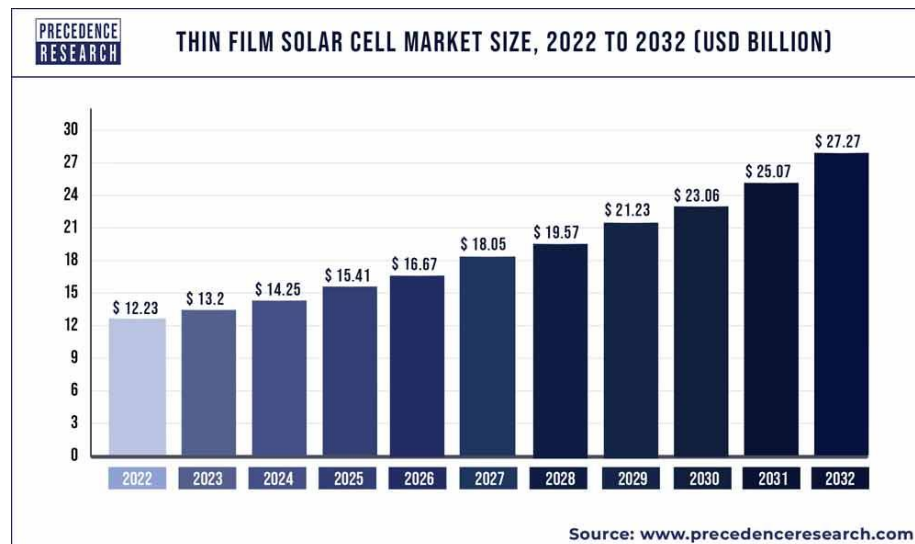


Figure1.5 The thin film solar cell market size from 2022 to 2032.

As shown, the solar cell market was valued at USD 12.23 billion in 2022 and is projected to reach approximately USD 27.27 billion by 2032, exhibiting a notable CAGR (Compound Annual Growth Rate) of 8.4% from 2023 to 2032 [11-13]. The leading thin-film technologies currently under development are:

1. Cadmium telluride (CdTe)
2. Copper indium (gallium) di selenide (CIGS or CIS)
3. Amorphous silicon (a-Si)
4. Organic thin-film PV (OPV)
5. Perovskites and tandem cells based on perovskites

Table 1.1 shows the most relevant properties of these semiconductor materials. CdTe stands out as a crucial category of thin-film PV semiconductors as it has the highest electron affinity. Its lowest melting point means that it can be deposited easily by physical vapor deposition techniques, especially the thermal evaporation method [14.15].

semiconductor	E_g (eV)	Electron affinity (eV)	Melting point (°C)
CdTe	1.45	4.28	1092
a-Si	1.12	4.05	1414
GaAs	1.42	4.07	1230
CZTS	1.45	4.50	
CIGS	0.97–1.43	4.07	1400

Table 1.1 shows the most relevant properties of the semiconductor PV materials.

1.4 CdTe THIN FILMS

CdTe is a highly promising photovoltaic material among the II-VI binary semiconducting compounds, particularly suitable for thin-film solar cell applications, achieving remarkable conversion efficiencies exceeding 22% and ranking as the second-lowest-cost material globally after silicon in the photovoltaic market [16-18]. Additionally, it is highly appealing due to its significant potential characteristics, including a high absorption coefficient ($5 \times 10^5 \text{ cm}^{-1}$) in the visible spectrum, leading to maximum photon absorption ($\geq 90\%$) for wavelengths $\lambda \leq 700 \text{ nm}$ in CdTe thin films with a thickness of merely 1-5 μm . CdTe has an optimal band gap (E_g) of 1.45 eV-1.50 eV, which aligns with the solar spectrum for maximum conversion efficiency [19–24,28]. Moreover, CdTe has high electrical resistivity, the ability to be an n-type or p-type semiconductor, and is chemically stable [21,24,29,30]. CdTe thin films can be grown in several ways, including thermal evaporation, RF magnetron sputtering, spray pyrolysis, pulsed laser deposition (PLD), molecular beam epitaxy (MBE), close-space sublimation (CSS), vapor transport deposition (VTD), metal-organic chemical vapor deposition (MOCVD), electro-deposition, and screen printing [17-19,21,31-35]. The global cadmium telluride market was valued at USD 7.6 billion in 2022 and is projected to attain approximately USD 20.28 billion by 2032, with a compound annual growth rate (CAGR) of 10% during the forecast period from 2023 to 2032, as shown in Figure (1.6) [36,37].

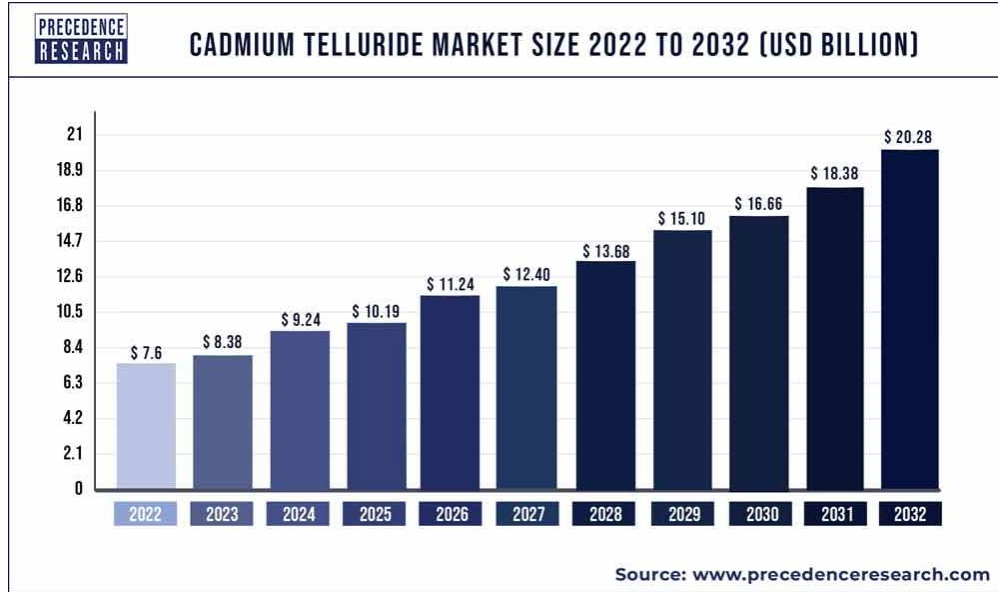


Figure 1.6 The CdTe market size from 2022 to 2032.

1.5 PLASMA

Materials experience phase transitions (solid $\rightarrow$ liquid $\rightarrow$ gas) when subjected to heating. Discontinuities characterize the phase transitions of a pure single-component substance and involve energy changes on the order of 10^{-2} eV per particle [39]. The prolonged heating of gas molecules results in an increase in the kinetic energy of each molecule. Particle collisions can displace electrons from atoms and molecules when a specific energy threshold is exceeded, producing various charged particles, including ions and electrons. The recombination of electrons and ions to restore the neutral molecule does not necessitate a threshold energy; however, it is significantly less probable. The energy necessary for converting a neutral gas into a mixture of ions and electrons can reach values between 1 and 30 eV per particle [39]. Plasma is a partially ionized gas composed of equal quantities of positively and negatively charged particles alongside neutral particles and

electromagnetic radiation [39]. The transition from gas to plasma occurs progressively with rising temperature and is not sudden. At ambient temperature, the proportion of ionized atoms is minimal, resulting in gases lacking plasma characteristics, as they typically do not conduct electricity and act as electrical insulators. The relative position of the states of matter with respect to temperature and the energy of the heavy particles is shown in Figure (1.7) [39].

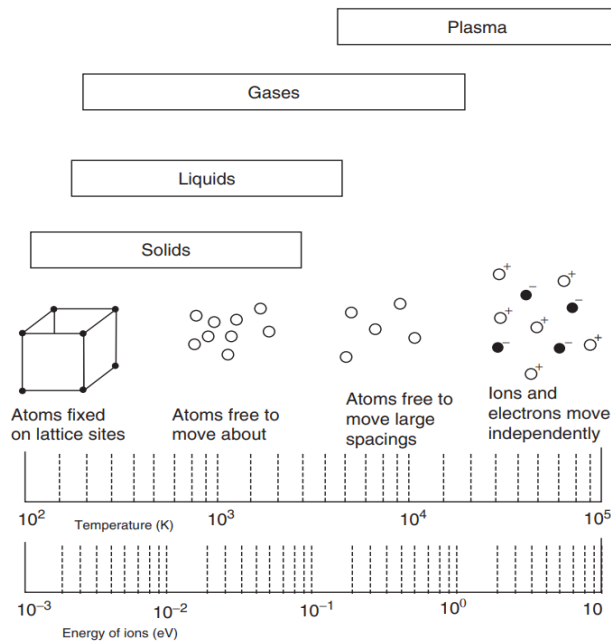


Figure 1.7 The relationship between states of matter, temperature, and the energy of dense particles.

The thermal heating of a gas in a container to generate plasma is impractical due to the absence of a solid container capable of enduring temperatures exceeding 4000 K. High energies can be imparted by generating charged particles in gas and exposing them to electric and magnetic fields. The charged particles are mobile and engage with one another via long-range electromagnetic forces. To prevent charged particles in the plasma from

contacting the container walls, it is necessary to confine them using appropriately configured electric and magnetic fields. As the plasma temperature increases, the confinement of plasma by these fields becomes exceedingly challenging [39]. The challenges arise from a minor disturbance in the plasma that can accumulate and lead to significant instabilities, similar to the disruption of traffic flow caused by a single disabled vehicle on the highway. The instabilities arise from the plasma's efforts to preserve internal charge neutrality and the interactions among plasma particles with varying velocities. These instabilities enable charged particles to collide with the plasma container's wall, dissipating their energy and potentially leading to the degradation of the intended plasma state or the container itself [39]. William Crookes (1879) examined the characteristics of gases under low pressure when subjected to electric fields to explore the structure of atoms and molecules [38,39]. He deduced from his experiments that electrified gases in a confined space, exhibiting uniform electrical and optical properties, manifest characteristics of a novel state of matter. Tonks and Langmuir (1931) introduced the term plasma to define the state of matter in a glow discharge, distinguishing it from conventional gases [38]. The energy supplied by an external power source is adequate to displace electrons from atoms and molecules, resulting in an equal quantity of oppositely charged particles [39]. The electrons are subsequently released from the atom and gain mobility. It was concluded that plasma attains a conductive state as charges are distinct entities capable of conducting current when exposed to electric and magnetic fields. The configuration of chambers can influence the distribution of ions and electrons in the plasma, while electromagnetic fields are employed to manipulate and energize them. In contrast to the potential energy of attraction between an electron and a proton in a neutral atom, the

potential energy of a typical plasma particle resulting from its neighboring particles is significantly less than its kinetic energy [38,39]. The presence of ions and electrons as distinct entities within plasma, interacting via forces, imparts several unique characteristics to plasma:

- Each charged particle in the plasma interacts simultaneously with all other charged particles, resulting in critical collective effects [39].
- Applied magnetic and electric fields can order and force charged particles to move regularly in cohesive groups [39].

1.6 PLASMA ETCHING PROCESS

Several applications of functional coatings necessitate surface activation via post-deposition treatments, which include thermal annealing, ion-beam modification, and plasma irradiation [40]. The properties of a plasma can be controlled by several parameters, such as the type of gas used, the input power mode (direct current, radio frequency, or microwave), energy, and pressure (which can range from 10^{-3} mbar to atmospheric pressure) [41]. Additionally, plasma process parameters include the gas flow rate and exposure time. The penetration depth of the ions in a plasma (typically, a few nanometers) characterizes this technique as a surface-modification method [41,42]. During plasma treatment, the ions break the chemical bonds between surface atoms and sputter the lighter atoms off the surface [42]. Hence, plasma treatment has emerged as a practical technique for modifying the physical and chemical properties of surfaces [42]. This is due to its simplicity, tuneability, low-temperature and low-pressure operation, the ability to conduct

it in-situ or ex-situ, reliability, energy efficiency, and relatively low surface damage [41,43-46]. Plasma treatment has been widely used to enhance the electrical conductivity of metal oxide thin films [43] through the creation and regulation of oxygen vacancies that contribute to enhancing the density of free-charge carriers [43,47]. Plasma treatment can also enhance surface crystallinity and stability, leading to increased carrier mobility, lower resistivity, and reduced trap density [47-49]. In addition, plasma-surface interaction can lead to defect formation, structural reconstruction, and band structure modulation [50]. Plasma treatment has been applied to a wide range of materials, including metal oxides [43,45,47-52], inorganic materials [53,50,41,42,44], and polymers [45,51] to enhance their applications in diverse fields including microelectronics fabrication [40,54], transparent conductors [41,43,51], photocatalysis [50,53], hydrogen production [50,53], photocurrent generation [53], and surface wettability [50,41]. Several gases have been used for the plasma activation of surfaces, including water vapor, hydrogen, oxygen, nitrogen, argon, helium, NH_3 , CO_2 , and CF_4 [41,46]. Of these gases, argon is usually selected because of its inertness, which does not introduce new chemical species on the treated surface [53,46,52], and its low etching rate [41]. Therefore, the role of argon is manifested in three forms:

1. Modification of the morphology of the surface [41],
2. Reduction of adsorbed oxygen species [51],
3. Breaking of the weak bonds between surface atoms, which modifies the surface chemistry [53].

As mentioned, CdTe-based technology has matured to an economically sustainable level compared to other photovoltaic materials. Nevertheless, several factors could be improved to enhance the efficiency of CdTe-based solar cells. One such factor is the synthesis of CdTe surfaces whose microstructure consists of grains with micro-sized lateral dimensions [55]. Plasma processing can modulate the surface microstructures with the possibility of enhanced lateral grain growth. Subsequently, the resulting enhancement in efficiency would reduce the manufacturing costs of CdTe photovoltaic modules [56].

1.7 LITERATURE REVIEW

Numerous efforts have been conducted to investigate the effects of plasma treatment on CdTe. It should be noted that post-deposition plasma treatment has many factors to consider, like the CdTe form, the type of gas, the duration of exposure to plasma, the type of plasma, and the ion energy. L. Svob *et al.*[57] investigated the impact of deuterium-plasma etching on cadmium telluride specimens. Etching was conducted at various temperatures ranging from room temperature to 350 °C. The etch sample rate of CdTe, as a function of r.f. power within the range of 10 to 45 W can be regarded as approximately linear. They determined that hydrogen plasma is effective for etching cadmium telluride, resulting in a clean and nearly stoichiometric surface. Vl. Kolkovsky *et al.*[58] investigated the electrical characteristics of defects in as-grown and hydrogen-plasma-treated CdTe thin films produced via molecular beam epitaxy. DC hydrogen plasma treatment was conducted at temperatures between 100 and 300°C for a duration of 1 hour, utilizing an applied DC

bias of 300 V. The hydrogen plasma treatment resulted in a substantial reduction of carrier concentration in the diode's depletion region. Furthermore, the carrier concentration diminishes with increasing temperature during the H₂ plasma treatment across all examined samples. A. Cimaroli *et al.*[59] investigated the impact of oxygen plasma treatment on the efficiency of CdTe thin film solar cells. They investigated the impact of oxygen plasma treatment on the fluorine-doped tin oxide (FTO) layer of commercial glass substrates and the cadmium sulfide (CdS) layer prior to the deposition of the cadmium telluride (CdTe) absorber layer. They evaluated the performance of the cells subjected to different plasma treatments in relation to the untreated cells. The researchers determined that the solar conversion efficiency was significantly enhanced following the plasma cleaning of the FTO, with additional improvement observed after the plasma cleaning of the CdS layer. S. Shanmugan *et al.*[40] investigated the impact of N⁺ ion bombardment on the characteristics of CdTe thin films. Thin films were bombarded with N⁺ ions at a voltage of 150 V and varying ion frequencies for approximately 10 minutes. They determined that a shift in the (111) peak positions corresponded to a variation of the lattice *d*-spacing. Additionally, they discovered a variation in crystallite size due to ions collisions. Furthermore, they determined that ion bombardment results in a decrease in transmittance near the fundamental absorption edge. D. E. Swanson *et al.*[60] investigated the impact of plasma cleaning on the TCO layer of a CdTe solar cell. The plasma-cleaning current density was empirically optimized to 0.11 mA/cm² for 30 seconds in a 200 mTorr N₂O₂ environment. Current-voltage (J–V), quantum efficiency (QE), electroluminescence (EL), and light-beam-induced-current (LBIC) measurements were employed to delineate the distinctions between plasma-cleaned and standard-cleaned devices. Limited research has been

conducted on the impact of argon plasma on CdTe, including studies on CdTe crystals. S. J. Pearton *et al.*[61] investigated plasma etching of ZnS, ZnSe, CdS, and CdTe in electron cyclotron resonance CH₄/H₂/Ar and H₂/Ar discharges. The standard total flow rates were 30 sccm. The process pressure fluctuated between 1 and 25 mTorr, the input power ranged from 100 to 500 W, and the radio frequency-induced direct current (DC) bias changed from -100 to -250 V. A. Sharmin *et al.*[62] studied the effect of argon pressure on the physical characteristics of cadmium telluride (CdTe) thin films by close-spaced sublimation. V. S. Shanmugan *et al.*[63] investigated the impact of Ar⁺ ion irradiation on the structural and optical characteristics of CdTe thin films deposited via electron beam evaporation. Following the preparation of thin films, they irradiated them with Ar⁺ ions for 10 minutes at three distinct ion flux densities. The researchers determined that an increase in the Ar⁺ ion flux resulted in a slight shift of the (111) crystalline line to a higher angular position. Furthermore, it was discovered that Ar⁺ ion irradiation enhances the bandgap of the synthesized CdTe thin films along with the structural parameters.

1.8 SCOPE OF WORK

This study involved the deposition of CdTe thin films via thermal evaporation, a method characterized by its simplicity, cost-effectiveness, high purity, and rapid deposition rate. The films underwent post-treatment in an argon plasma, and their properties were assessed to elucidate the plasma's effect on the photoresponse of these films. The primary challenge in our work is identifying the appropriate parameters to improve the photoresponse of CdTe thin films. This study is the first to utilize Ar plasma for one hour on CdTe thin films, in contrast to prior research. The resultant structure, morphology, and chemical properties of the CdTe films were examined. Furthermore, the optical and electrical characteristics of the resultant films were assessed.

CHAPTER 2

EXPERIMENTAL TECHNIQUES

2.1 PHYSICAL VAPOR DEPOSITION

Physical vapor deposition (PVD) encompasses various vacuum deposition techniques for the creation of thin coatings and films. In PVD, a material is transformed from a condensed phase to a vapor phase and subsequently reverts to its original phase as a thin film. The PVD process is illustrated in Figure 2.1. The principal processes in PVD are evaporation and sputtering. It is utilized in the fabrication of products necessitating thin coatings for optical, mechanical, electronic, or chemical functions. These coatings exhibit durability owing to their corrosion resistance, increased hardness, and elevated impact strength [64]. In PVD, the material intended for deposition is vaporized from the solid state. The vapor arrives at the substrate by traversing a low-pressure zone (vacuum), followed by the deposition of a film on the substrate. The atoms arriving at the growing surface are essential for the film's structural development, necessitating a balance between the arrival rate and surface mobility to allow atoms sufficient time to reach equilibrium sites [65].

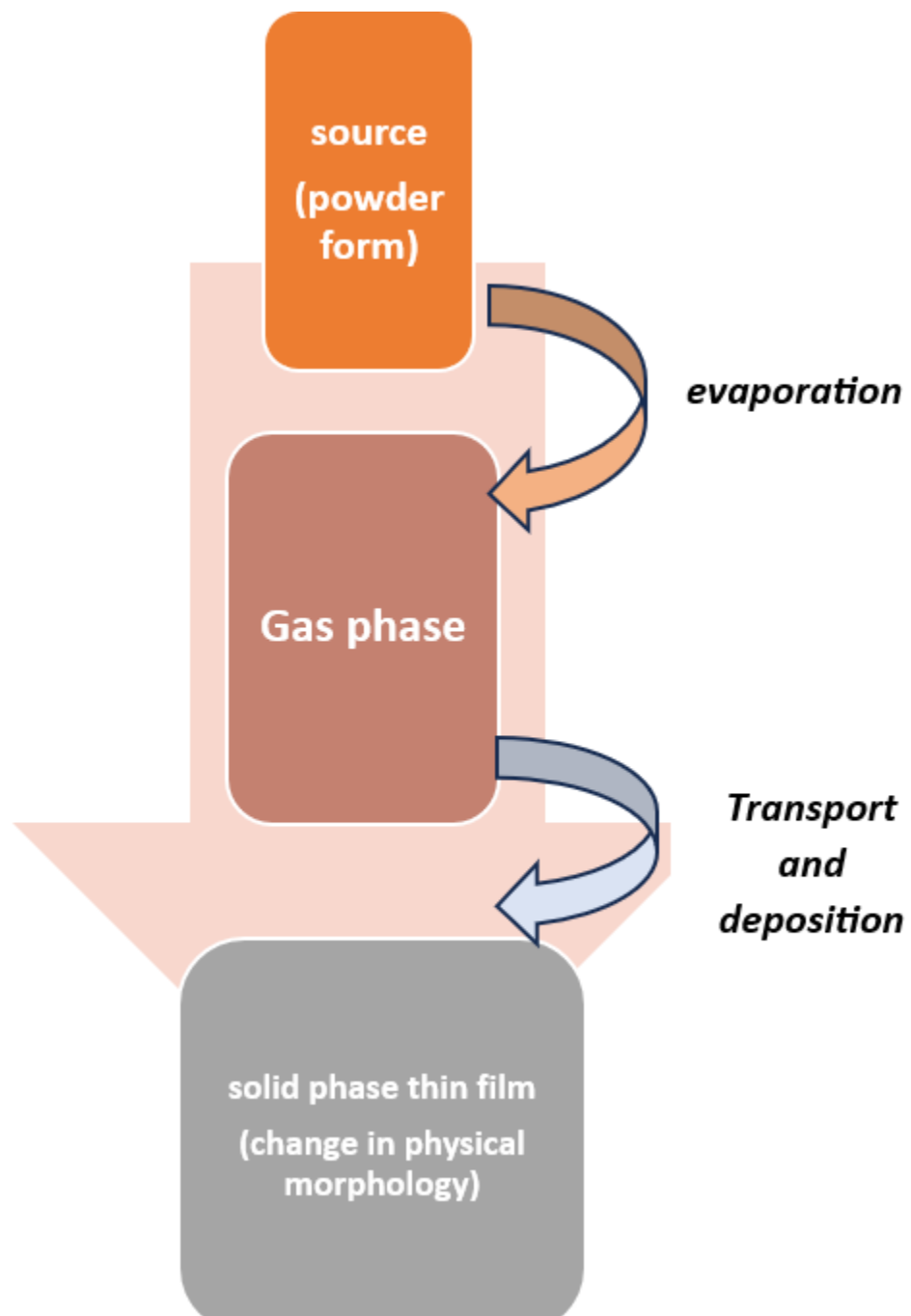


Figure 2.1 A schematic diagram of the PVD process.

One of the most common PVD techniques is thermal evaporation. Thermal evaporation is a recognized technique for applying a thin coating, wherein the source material vaporizes in a vacuum due to elevated temperatures. This enables vapor particles to travel and directly deposit onto a substrate, where they subsequently solidify. To achieve high temperature, a direct current (DC) is applied to a boat containing the source in powder form. Heating the material until it exceeds its melting point leads to evaporating it to appear as a vapor flux directed to be deposited on the substrate. This process must occur in a high vacuum that reduces the collisions with ambient gas molecules in the space between the source and substrate. Also, gaseous contamination is reduced as there will be no molecules other than the species we are vaporizing. A diagram of a thermal evaporation system is presented in Figure (2.2).

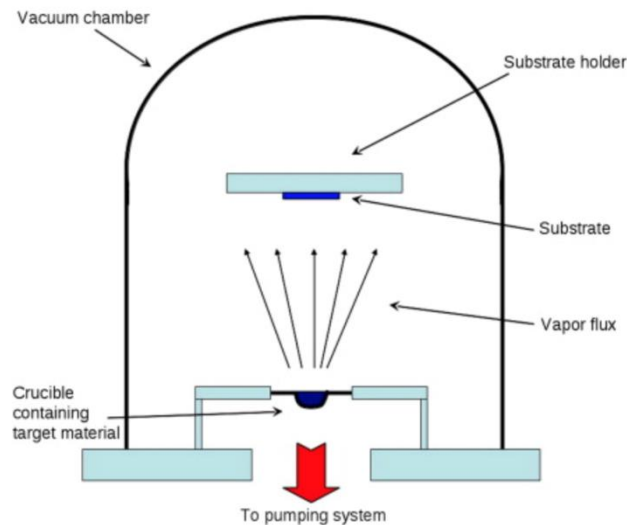


Figure 2.2 Schematic diagram of thermal evaporation

To understand the importance of the thermal evaporation deposition technique, some of its applications can be mentioned. Thermal evaporation deposition serves multiple applications, including the following: Thermal evaporation deposits metal contacts, gate materials, and barrier layers in IC manufacturing [67]. It can also be used in display technologies, especially for liquid crystal displays (LCDs) and organic light-emitting diode (OLED) screens [67-69]. It deposits thin films of materials like cadmium telluride (CdTe), copper indium gallium selenide (CIGS), or perovskites for photovoltaic cells. It also deposits organic and metallic layers for the construction of OLED displays used in smartphones, TVs, and other screens. Also, it can be used to coat optical mirrors, filters, and anti-reflective coatings in laser systems and optical devices. Thin films of magnetic materials, such as iron and cobalt alloys, are deposited on hard drives and other storage devices. It deposits metal and dielectric layers in CD, DVD, and blu-ray discs. It is used in the deposition of thin layers of metals or metal oxides, like titanium nitride or aluminum oxide, to improve the hardness, wear resistance, and corrosion resistance of tools, medical implants, and aerospace components. It is used to deposit thin films of semiconducting oxides or metals for gas detection applications [67-70]. Thermal evaporation is used to create thin films that can be further patterned into nanostructures, such as quantum dots, nanowires, and other nanoscale devices [67]. It also can be used in the deposition of thin metallic films to study surface plasmon resonance, used in sensors and photonic devices. It is used also in thin-film coatings on electrodes for enhancing capacitance and energy density in energy storage devices [67,69]. The reason why thermal evaporation is one of the most favorable methods for depositing a thin film is that it has many advantages, a notable advantage of thermal evaporation is its comparatively low cost relative to other

deposition methods [67-70]. The apparatus necessary for thermal evaporation, including resistive heating elements or electron beam sources, is typically less complex and more economical than that needed for alternative techniques, such as sputtering. The streamlined equipment configuration leads to reduced installation and maintenance expenses, rendering thermal evaporation a favored option for both small-scale research laboratories and large-scale industrial production. Moreover, the procedure generally necessitates fewer consumables and entails a simple operational configuration, thereby further diminishing the total production expenses. Thermal evaporation is valued for its elevated deposition rates and efficiency [67-70]. In contrast to more complex methods that may require numerous processing stages or gas-phase reactions, thermal evaporation can deposit thin films at comparatively high rates, particularly when elevated temperatures are employed. This efficiency renders it appropriate for extensive production settings where time is a crucial element. Elevated deposition rates allow manufacturers to rapidly create uniform films on diverse substrates, which is crucial for industries necessitating rapid turnaround times, such as semiconductor fabrication and optics [67,70]. A further benefit of thermal evaporation is the elevated purity of the resultant films. The process occurs in a vacuum environment, resulting in negligible contamination from atmospheric gases or other environmental influences [67-70]. The source material is generally vaporized without chemical reactions, minimizing the risk of impurities being integrated into the film. Thermal evaporation exhibits significant versatility regarding the materials it can deposit. It is compatible with various materials, including metals, dielectrics, and organic compounds. The technique provides consistent coating on complex substrate geometries. Thermal evaporation depends on vaporized atoms uniformly condensing on a cooler

substrate, enabling consistent thickness on three-dimensional or irregularly shaped objects. A further advantage is the negligible damage to substrates resulting from the comparatively low kinetic energy of the evaporated atoms [67-70].

2.2 THIN FILM DEPOSITION AND PLASMA IRRADIATION

The experimental procedure is shown schematically in Figure (2.3).

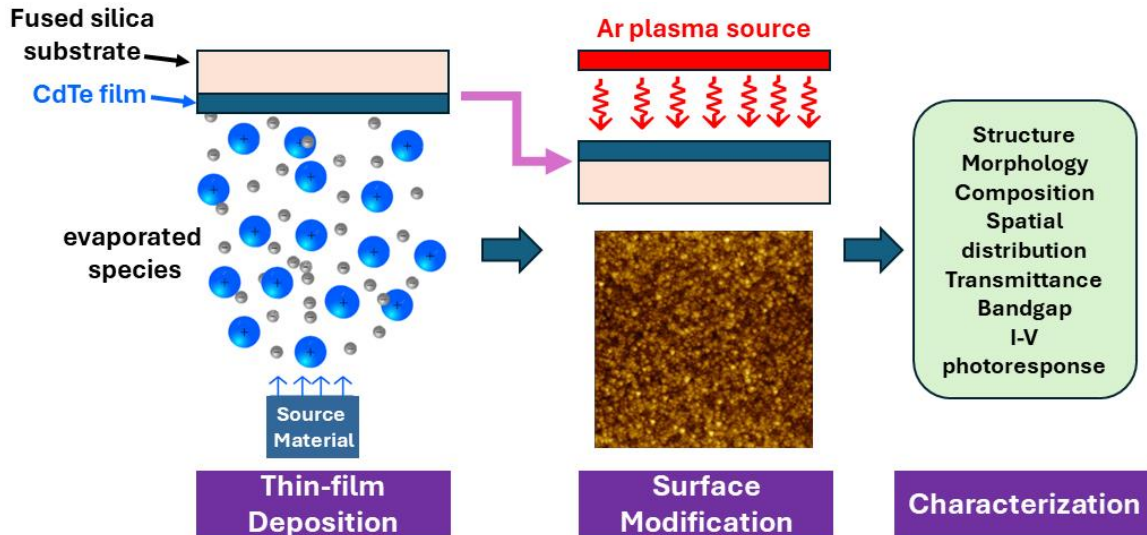


Figure 2.3 The experimental procedure: First, the films were deposited by thermal evaporation. Then, they were irradiated with the plasma source. Finally, various characterizations were used to assess plasma's influence on the films' properties.

The initial substance used was a CdTe powder with a 99.99% purity level. It was subjected to thermal evaporation from a molybdenum boat within a Leybold L560 box coater. The films were deposited on fused silica substrates, which were cleaned before deposition using a special optical-grade detergent and isopropanol. The deposition system was initially pumped down to a base pressure of 2×10^{-5} mbar, while the pressure increased to 8×10^{-5} mbar during evaporation. Although the substrates were not intentionally heated, the

substrate temperature increased by no more than 50 °C during evaporation due to the process's thermal energy. The distance from the source to the substrate was 40 cm, and the substrates were rotated during evaporation to ensure uniform film deposition. The deposition rate was 1.33 nm/s. Following deposition, the films underwent plasma irradiation using an Oerlikon Univex 350 magnetron sputtering system Figure (2.4). The system was initially evacuated to 5×10^{-6} mbar. The ion plasma source was argon gas (purity 99.99%) applied at flow rates of 20, 30, and 40 standard cubic centimeters per minute (sccm) under DC powers (P) from 20 to 80 W. The chamber pressure during plasma irradiation was 2.5×10^{-2} mbar. The plasma treatment was carried out for 1 hour. As a result of the plasma irradiation, the substrate temperature increased from 75 °C to 135 °C as shown in Table (2.1).



Figure 2.4 Oerlikon Univex 350 magnetron sputtering system.

Ar Flow Rate (sccm)	Power (%)	Voltage (KV)	Current (mA)	Power (W)	Temperature (°C)
As deposited	...	...	...	...	...
20	40	1.6	12	20	75
20	50	2.1	14	30	98
20	60	2.2	18	40	125
30	40	1.3	32	42	113
30	50	1.3	34	45	115
30	60	1.3	36	47	119
40	40	1.0	44	45	130
40	50	1.4	42	60	132
40	60	2.0	40	80	135

Table 2.1 The plasma working parameters.

As shown, nine samples in addition to the reference sample (As deposited) were studied. At first, the power was selected as a percentage value, then due to the change in argon flow rate, and the percentage of plasma power led to a change in the current and voltage which helped in calculating the power as a value in watt by using the equation:

$$P = IV \quad (2.1)$$

This concept will be suitable for use instead of the percentage value. So that, in all the results, the effect of increasing power at each value for the argon flow rate will be studied.

2.3 CHARACTERIZATION TECHNIQUES

Different characterization techniques were used to investigate the properties of the films. These are mentioned below.

2.3.1 THICKNESS MEASUREMENT

The thickness of the films was assessed utilizing a stylus surface profilometer (KLA Tencor D-500), as shown in Figure (2.5). The KLA stylus-based surface profilometer is a computerized, high-sensitivity instrument that quantifies surface flatness, curvature, roughness, waviness, and step height across diverse applications. The profilometer quantifies vertical depths and horizontal widths by employing a probe tip that is gradually traversed across a sample surface. It is positioned on a vibration isolation table to minimize noise, which can impact the minimum height and resolution. The scan analysis can be conducted utilizing software that measures profiles, roughness/waviness, and step height.

Working Principle: The profilometer measures film thickness by gliding a 2-micron radius diamond stylus across the surface of a horizontally positioned substrate. The motion of the stylus in the vertical axis is measured to within tens of Ångstroms. An HD video microscope with keystone correction is used to help position the stylus in the desired scan location, with adjustable magnification, lamp brightness, image brightness, and contrast. After taking a scan, a 2-D graphic representation of the profile of the film is shown on the screen. The scan may be leveled between any two desired points on the profile, and then measurements can be taken by positioning two cursors, with one cursor on the bottom

of the step and one on the top of the step. The cursors have an averaging feature that allows to spread out the point across any desired range of data, to calculate an average for that range. This averaging feature is very helpful in canceling out background noise, and it allows for more accurate step height measurements and more accuracy during the leveling procedure. The stylus pressure is digitally adjustable from 0.03 mg to 15 mg. The maximum scan thickness range in the vertical axis is 1200 microns. The scan speed is adjustable from 0.04 mm to 0.4 mm per second. The maximum scan length is 30 mm.

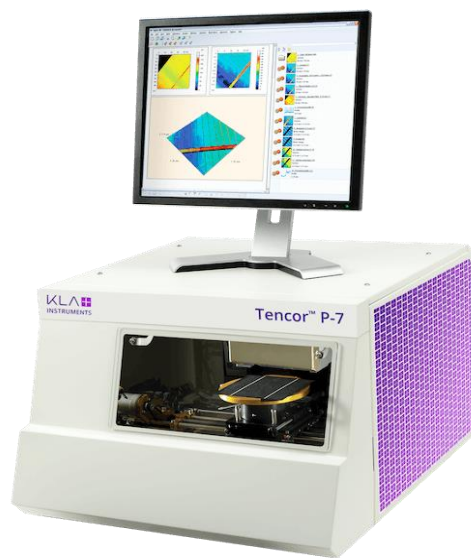


Figure 2.5 KLA Tencor D-500 profilometer.

2.3.2 Structural Analysis

X-ray diffraction (XRD) is an essential technique for obtaining atomic-scale data from crystalline materials. It is employed to define the crystallographic structure, crystallite size, and preferred orientation in solid samples.

Operational Principle: X-ray diffraction functions in accordance with Bragg's law. When a monochromatic X-ray beam of wavelength λ strikes a crystalline material at an angle θ , the path length traveled by rays reflected from successive planes must differ by an integer n of wavelengths.

$$n \lambda = 2 d \sin \theta \quad (2.2)$$

where d is the space between the atomic planes of the sample. The schematic diagram in Figure (2.6) illustrates the geometry of Bragg's law. The diffracted waves interfere constructively, mirroring the precise symmetry found in atomic distribution. The diffracted X-rays are subsequently detected, processed, and quantified to ascertain the atomic distribution within the material. The structural properties of our films were analyzed using a Bruker D₂ Phaser diffractometer. This utilized a Cu (K_α) source (with a wavelength of 1.54 Å), operated at 30 kV, and employed a scanning step size of 0.01°.

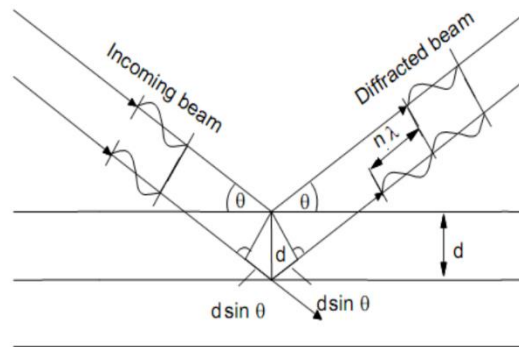


Figure 2.6 Schematic diagram of Bragg's law of diffraction.

2.3.3 Surface Morphological Analysis

Atomic force microscopy (AFM) is a high-resolution scanning probe microscopy method that achieves resolution on the order of fractions of a nanometer at a material's surface.

Operational Principle: The Atomic Force Microscope quantifies intermolecular forces and visualizes atoms by probing the specimen's surfaces at the nanoscale [73]. Its operation is facilitated by three principal mechanisms: Surface sensing, detection, and imaging. The (AFM) conducts surface sensing utilizing a cantilever, a rigid element resembling a beam or plate that extends perpendicularly from support, forming a vertical connection akin to a wall. The cantilever possesses a pointed tip that traverses the sample surface by generating an attractive force as it approaches the surface [73]. As it approaches the sample surface, a repulsive force increasingly dominates, causing the cantilever to deviate from the surface. As the cantilever deflects away from the sample surface, the direction of the reflected beam alters, and a laser beam identifies this deviation by reflecting off the flat surface of the cantilever. Utilizing a position-sensitive photodiode (PSPD—a device founded on silicon PIN diode technology employed to ascertain the position of the integral focus of an incoming light signal), it monitors alterations in deflection and direction of the reflected beam and documents them. (AFM) captures the surface topography of a sample by scanning a cantilever across a designated area using force [73]. The elevation of the sample's surface influences the beam's deflection, which is monitored by the Position-sensitive photodiode (PSDP). The microscope features a feedback loop that regulates the cantilever tip's length just above the sample surface, thereby maintaining the laser position and producing an accurate imaging map of the surface [73]. A schematic diagram of an atomic force microscope is shown in Figure (2.7).

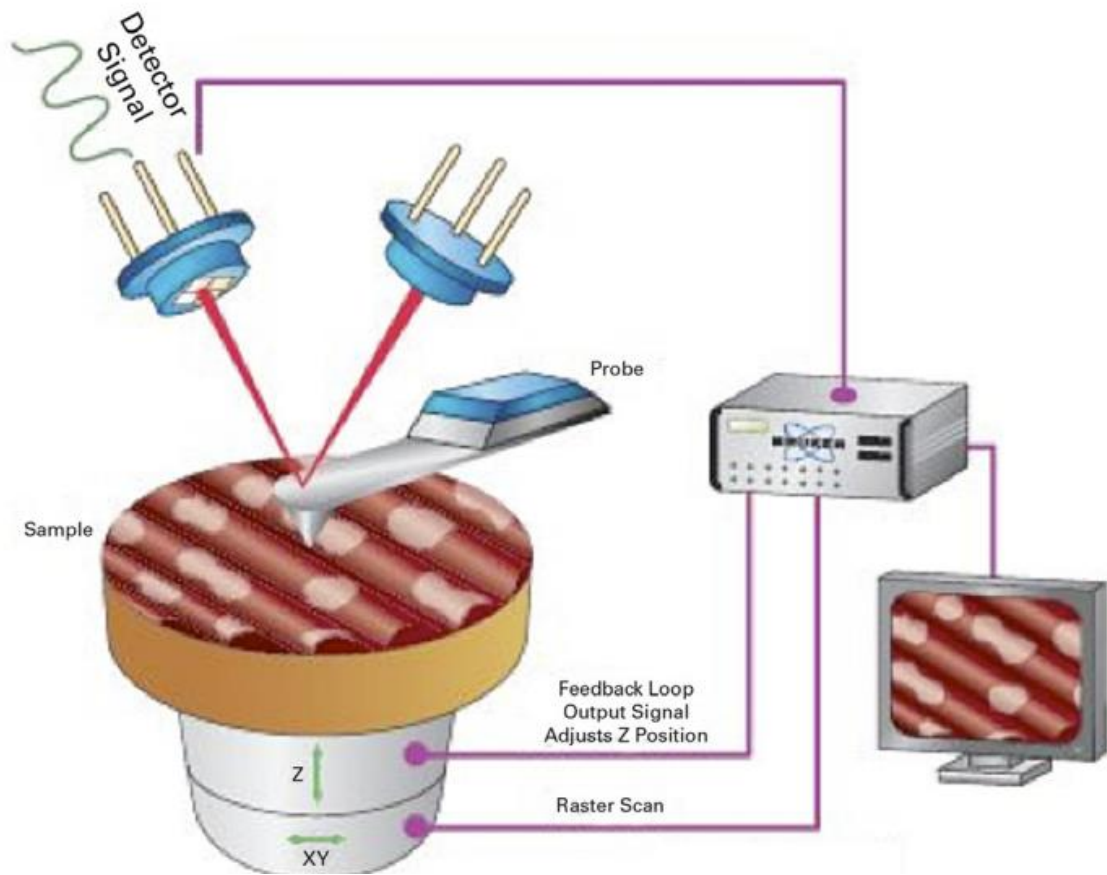


Figure 2.7 Schematic diagram of the working principle of an atomic force microscope.

2.3.4 CHEMICAL COMPOSITION

The chemical composition of the films was determined using two techniques:

A- X-RAY PHOTOELECTRON SPECTROSCOPY

X-ray photoelectron spectroscopy (XPS) is a technique for surface chemical analysis employed to examine a material's surface chemistry. It offers comprehensive information regarding the chemical composition of the surface, typically to a depth of 1-10 nm.

Working Principle: (XPS) is a surface analysis method that relies on the photoelectric effect as shown in Figure (2.8) [74]. Every atom on the surface possesses core electrons with a binding energy that is conceptually, though not strictly, equivalent to the ionization energy of that electron. When an X-ray beam is aimed at the sample surface, the energy of the X-ray photon is entirely absorbed by the core electron of an atom [74]. When the photon energy is sufficiently high, the core electron will escape from the atom and be emitted from the surface. The electron emitted is termed the photoelectron. The binding energy E_b of the core electron is defined by the Einstein equation [74]:

$$h\nu = E_b + E_k + \phi \quad (2.3)$$

$$E_b = h\nu - (E_k + \phi)$$

where $h\nu$ is the X-ray photon energy; E_k is the kinetic energy of the photoelectron, and ϕ is the work function of the spectrometer (about 4~5 eV). From the binding energy and intensity of a photoelectron peak, the elemental identity, chemical state, and quantity of an element are determined.

For our films, XPS was performed in a Thermo Scientific Escalab 250Xi spectrometer, which is fitted with a monochromatic Al K α X-ray source and has a 0.5 eV instrumental energy resolution. Prior to the XPS analysis, the samples were transferred in air to the XPS analysis chamber. The C1s peak of hydrocarbon contamination, at a binding energy of 284.5 eV, was used as an energy reference. During the XPS analysis, the samples were maintained at ambient temperature at a pressure of 5×10^{-7} Pa.

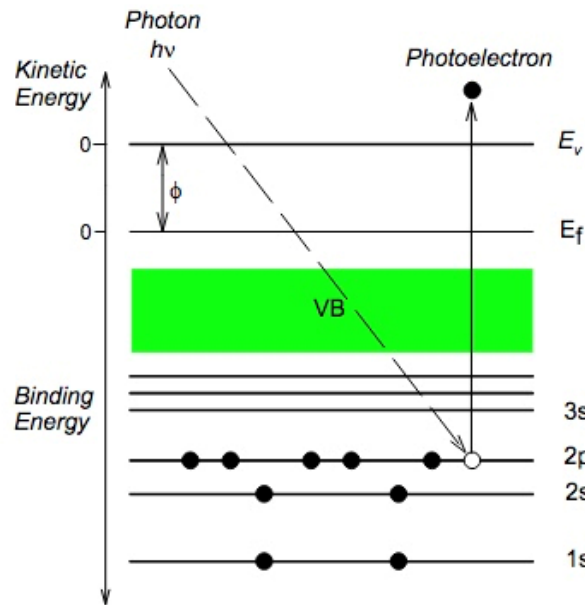


Figure 2.8 Schematic diagram of the basic principle of X-ray photoelectron spectroscopy.

B- Time-of-flight secondary-ion mass spectrometry (TOF-SIMS)

Time-of-flight secondary-ion mass spectrometry (TOF-SIMS) is, like XPS, a technique for surface chemical analysis employed to examine in-plane and in-depth chemistry. It offers chemical information to a depth of 1–2 nm and deeper if depth profiling is employed.

History of SIMS/TOF-SIMS: The first SIMS instruments were built in the late 1940's by Herzog and co-workers. The SIMS methodology has been developed and exploited mainly for semiconductor (microelectronics) requirements. During the late 1960's Benninghoven and co-workers (University of Münster) introduced a variant which set out to perform monolayer analysis by using conditions which cause negligible surface damage. This approach is known as static SIMS because the surface under investigation remains practically undamaged. The introduction of the TOF analyzer (i.e. TOF-SIMS) has outperformed the magnetic and quadrupole-based SIMS instruments.

Working principles: A solid surface is bombarded by primary ions of high kinetic energy (keV range) Figure 2.8. The primary ion energy is transferred to target atoms via atomic collisions and a so-called collision cascade is generated. Part of the energy is transported back to the surface allowing surface atoms and molecular compounds to overcome the surface binding energy. The kinetic energy of the secondary ions are ~20 eV. The size corresponds to atoms, molecules, fragments, and clusters. The escape depth is 1–2 nm.

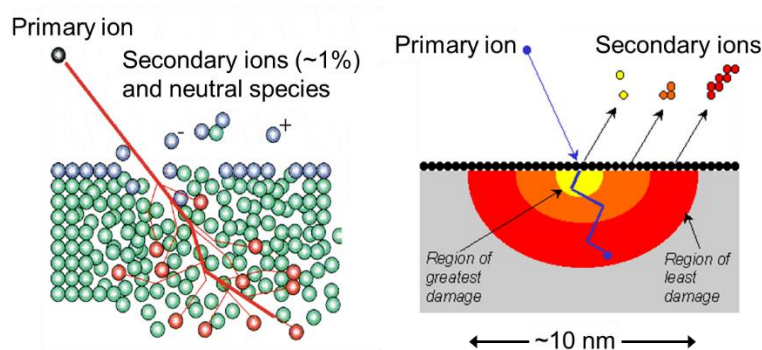


Figure 2.9 shows the working principle of TOF-SIMS.

After the ions have been expelled from the surface, they are accelerated to having the same kinetic energy (~ 2 keV) and left to drift through a time-of-flight (TOF) analyzer. On the way to the detector, the secondary ions are reflected via an electronic mirror that has focusing properties. Since the secondary ions have the same kinetic energy they will have different velocities according to their mass, that is, the lighter ones will be faster and thus hit the detector quickest. The time-of-flight can then be converted to mass, which produces a mass spectrum. Since time-of-flight is measured it must be pulsed ionization. In this work, TOF-SIMS measurements were conducted utilizing a TOF-SIMS M6 (ION-TOF GmbH, Münster, Germany). The schematic diagram is illustrated in Figure (2.9). The ion images were acquired using a primary ion beam of 30 keV Bi_3^+ , resulting in a target current of 0.3 pA. Stage rastering was utilized over a surface area of 10×10 mm² employing 20×20 (400) patches, with 1 frame per patch, 20 shots per frame, and in interlaced mode. The data was aggregated by a factor of 4, resulting in an overall image dimension of 640 by 640 pixels. The primary ion beam parameters employed to acquire the depth profiles were 30 keV Bi^+ , yielding a target current of 1.0 pA. A 300×300 μm^2 area was examined, centered

within a sputter crater measuring $600 \times 600 \mu\text{m}^2$. The sputter ion beam parameters employed for acquiring the depth profiles were 20 keV Ar_{1863}^+ , yielding a target current of 14 nA. Ion imaging and depth profiling were conducted exclusively in positive ion mode with a beam diameter of approximately $5 \mu\text{m}$. A flood gun was employed to reduce the accumulated charge on the surface. Desorbed secondary ions were accelerated to 2 keV, subjected to mass analysis in the flight tube, and subsequently accelerated to 10 keV prior to detection. Data analysis was conducted utilizing the software package Surface Lab (version 7.3.130058).

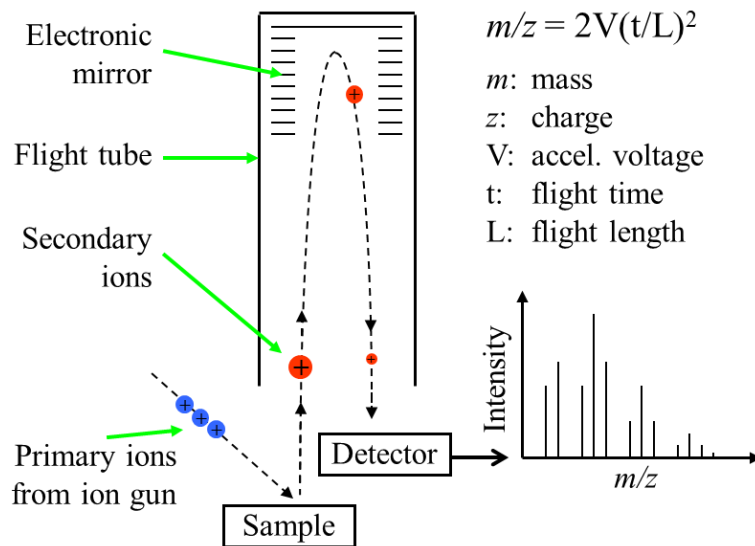


Figure 2.10 The schematic diagram of the principle of TOF-SIMS.

2.3.5 OPTICAL MEASUREMENTS

The optical properties of a material utilized as a thin film are ascertained via optical measurements. The optical measurements provide data on the band gap and the optical constants of the films. The interaction of light with a thin film is depicted in a schematic diagram in Figure (2.10). When interacting with the thin film, a fraction R of the radiation is reflected from the film's upper surface, while a fraction T is transmitted through the film. A fraction of the radiation is absorbed A by the films and a fraction S is scattered because of surface and volumetric imperfections. The total of all contributions is usually articulated as

$$R + T + A + S = 1 \quad (2.4)$$

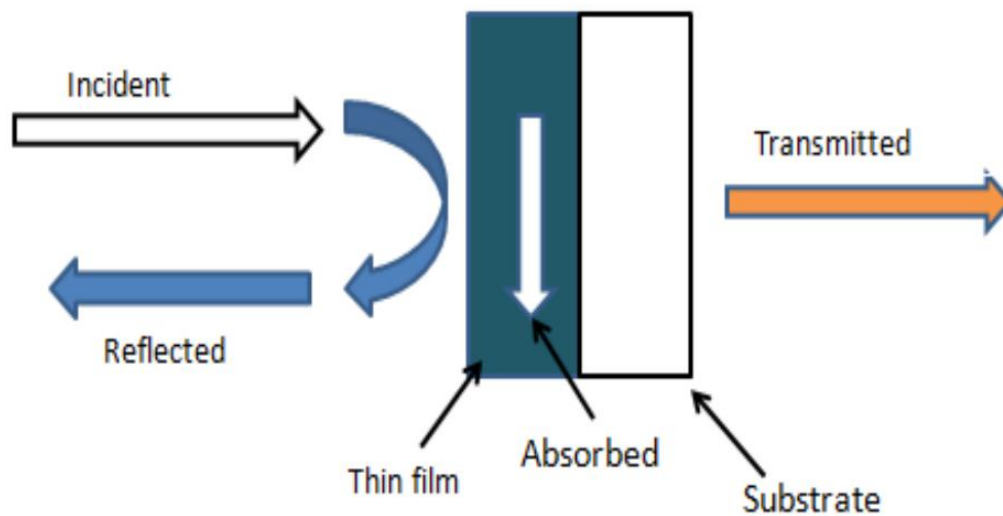


Figure 2.11 Basic principle of optical measurements of a thin film.

In our work, normal-incidence transmittance T was measured over a wavelength range of 700–1700 nm using a Jasco V-570 double-beam spectrophotometer Fig(2.11) [75].

A spectrophotometer is an instrument employed to measure light intensity as a function of wavelength. A double-beam spectrophotometer evaluates light intensity across two optical pathways, one housing a reference beam and the other containing the test sample. The spectrophotometer consists of light sources, diffraction gratings, filters, photodetectors, and signal processors. The light source emits wavelengths across the visible, ultraviolet, and near-infrared spectra. The filters and diffraction grating separate light into its wavelengths, enabling a specific range of wavelengths to be directed onto the sample. The photodetector converts incident light into electrical current, which is then relayed to the signal processor. The signal processor converts current into absorbance, transmittance, and reflectance values, which are then relayed to the display. The spectrophotometer quantitatively evaluates the amount of light transmitted through both the reference beam and the sample during transmission measurements [76].

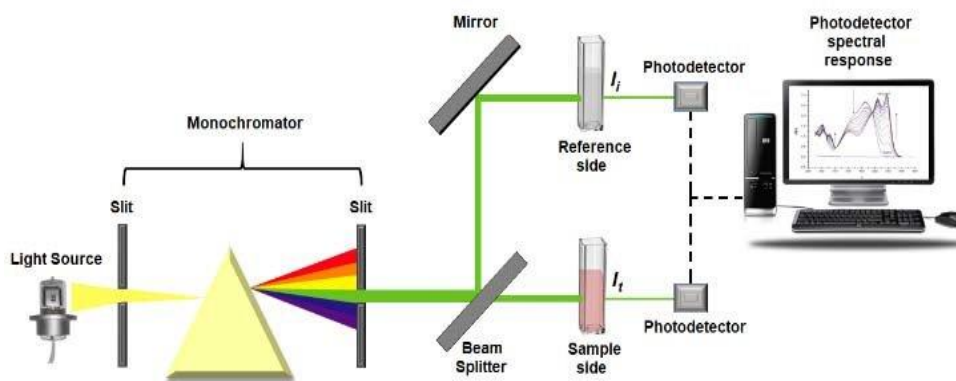


Figure 2.12 Schematic diagram of a double-beam spectrophotometer.

2.3.6 ELECTRICAL MEASUREMENTS

Electrical measurements can reveal the electrical properties of a material deposited as a thin film. These measurements can also provide information about the films' resistivity and current-voltage (I-V) characteristics. This information can be extracted using the two-probe method, which is demonstrated in a schematic diagram in Figure (2.12).

Working principle: Two parallel gold electrodes were deposited on films by thermal evaporation (length: 2.5 cm, spacing: 2 mm). Then, the I-V curves were obtained using a longitudinal configuration by sweeping the voltage from 0 to 10 V and measuring the corresponding current using a Keithley 238 source measure unit (SMU). All measurements were conducted in no-light conditions and with illumination provided by a 75-W xenon lamp. For photocurrent measurements, the light was directed perpendicular to the surface of the film within the gap between the two gold electrodes.

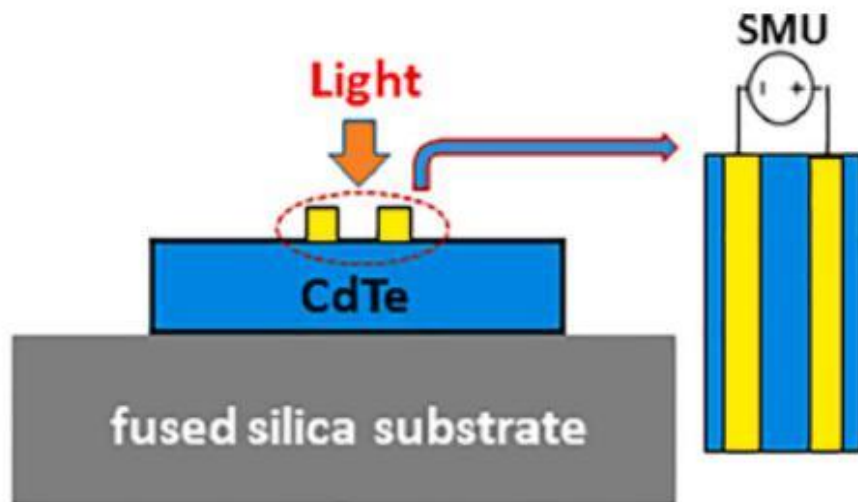


Figure 2.13 schematic diagram of the two-probe method.

CHAPTER 3

STRUCTURAL PROPERTIES

3.1 THICKNESS MEASUREMENTS

Table (3.1) shows the thicknesses of the films measured by the stylus surface profilometer. The as-deposited films had a thickness of 1654 nm, corresponding to a deposition rate of 1.33 nm/s. Such a deposition rate is relatively high and shows the efficiency of thermal evaporation in depositing highly thick films in a relatively short time. By plotting a relation between the plasma power and the film thickness, Figure (3.1), there was a substantial reduction in thickness, which was proportional to plasma power, due to the etching effect of the plasma.

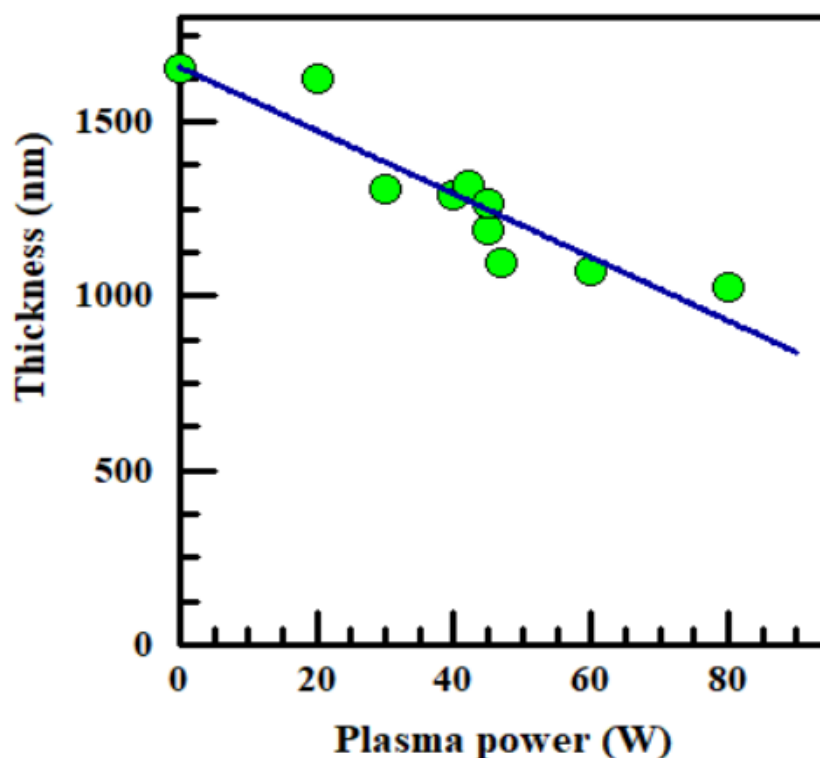


Figure 3.1 Variation in the thickness of the films with plasma power.

3.2 X-RAY DIFFRACTION (XRD) ANALYSIS

Figures (3.2-3.4) show the XRD patterns for the CdTe films before and after plasma treatment at plasma powers ranging from 20 W to 80 W. Identification of material phases and peak assignments was based on data from the International Center for Diffraction Data (JCPDS). The films exhibited a polycrystalline structure with peaks along the (111), (220), and (311) crystallographic directions of the cubic zinc blende structure of CdTe centered at angles $2\theta = 23.72^\circ$, 39.21° and 46.34° , with dominant growth along the (111) direction. To ensure the cubic structure of the films this relation is used.

$$\frac{4\sin^2\theta_{hkl}}{\lambda^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (3.1)$$

where $\lambda = 1.5406 \text{ \AA}$ is the wavelength of the X-rays, and a is the lattice constant ($= 6.48 \text{ \AA}$) [77]. At lower plasma power levels (20 W), the peak intensities closely resembled those of the films in their as-deposited state. Treatment with higher plasma powers $> 20 \text{ W}$, increased the XRD intensity. The change in the peak's intensity can be ascribed to defect creation, chain scission and formation of free radicals. It may be attributed to the plasma's thermal energy which results in forming a new and ordered structure after ion irradiation [78,79]. Also, the etching of the surfaces by plasma leads to the etching of some external particles, and that means there will be more particles to scatter X-ray. As the plasma power increased ($P \geq 30 \text{ W}$), an additional peak appeared along the (200) direction centered at $2\theta = 28.22^\circ$, which is an indication that the thermal energy provided by the plasma was sufficient to overcome the minimum energy density for crystalline growth along this direction. The crystalline quality of the films was assessed by six parameters: crystallite

size, texture coefficient, orientation factor, interplanar spacing, lattice constant, and strain.

The crystalline size was estimated using the Scherrer equation [80]:

$$L = \frac{K \lambda_{\alpha}}{\beta \cos \theta} \quad (3.2)$$

where K is a shape factor taken to be unity, β is the full width at half maximum (FWHM) of the (111) XRD peak, and θ is the corresponding Bragg angle. The FWHM and the crystalline sizes are generally affected by plasma power. The texture coefficient C_{hkl} for crystalline growth along the (hkl) direction is given by the equation [81]:

$$C_{hkl} = N \frac{I(hkl)/I_o(hkl)}{\sum I(hkl)/I_o(hkl)} \quad (3.3)$$

where the summation is over the number of orientations ($N = 3$ or 4) considered, $I(hkl)$ is the measured relative intensity along the (hkl) direction of the films and $I_o(hkl)$ is the relative intensity along the same direction in bulk CdTe [JCPDS file no. 15-0770]. The orientation factor (f) is defined as [81]:

$$f = \sqrt{\frac{\sum (C_{hkl} - 1)^2}{N}} \quad (3.4)$$

The interplanar spacing (d_{hkl}) along the (hkl) direction is related to the diffraction angle (θ) via the Bragg's law [82]:

$$d_{hkl} = \frac{\lambda_{\alpha}}{2 \sin \theta} \quad (3.5)$$

For a cubic structure, the interplanar spacing for a given crystallographic direction (hkl) is related to the lattice constant (a) by the equation [82]:

$$a = \sqrt{h^2 + k^2 + l^2} d_{hkl} \quad (3.6)$$

which becomes $a = \sqrt{3} d_{111}$. The strain (ϵ) in the films was estimated using the equation [83]:

$$\varepsilon = \left[\frac{(d_{111})_f - (d_{111})_B}{(d_{111})_B} \right] \times 100 \quad (3.7)$$

where $(d_{111})_f$ and $(d_{111})_B$ are the values of the interplanar spacing along the (111) direction for the film and bulk material, respectively. The values of the structural parameters of the films ($d, C_{111}, C_{220}, C_{311}, C_{311} L, f, (d_{111})_f, a$, and ε) are listed in Table (3.1). For the lowest power ($P = 20$ W), plasma treatment reduced crystallite size, which may be attributed to the generation of defects upon ion bombardment [40] and the breaking of grains into smaller sizes [51]. However, as the plasma power increases to 30 W, smaller grains start to coalesce to form larger grains due to the higher thermal energy (effectively annealing) provided by the plasma [40]. At a power 40 W, the thermal energy is no longer sufficient to overcome the defect density generation by the plasma [40], and the crystallite size decreases to a value equal to that of the as-deposited film. Then, the crystalline size started to increase again due to the increase in the plasma power, which means that thermal energy is sufficient to overcome the defect density generation by the plasma. At 80 W, the crystalline size reaches its maximum value, which is considered more than double the value of the crystalline size of the as deposited one. The texture coefficients were evaluated for the four orientations to examine the degree of orientation. A value of $f = 0$ corresponds to a completely randomly oriented structure. As f increases, the degree of orientation increases. The film, which is treated by plasma at 30 W, is well-oriented as it has the maximum value of the orientation factor. The reduction in the lattice constant (and hence the interplanar spacing) for the plasma-irradiated film may be due to the smaller size of the ionic radius of argon compared to those of cadmium and tellurium [81,51]. This enables the Ar^+ ions to segregate in the CdTe lattice, shortening the bond between the Te^{2-} and Cd^{2+} ions and, thus, reducing the lattice constant [81,51]. Moreover, the (111) peak of the bulk

CdTe occurs at $(2\theta)_{111} = 23.76^\circ$, and the (111) peak of the as-deposited films occurred at $2\theta = 23.71^\circ$ which is lower than the bulk value. This indicates the presence of compressive stress in these films (positive strain), which could arise from the lattice mismatch between the CdTe thin films and the amorphous substrate [40,41]. On the contrary, the 2θ values of the (111) peak increased in the plasma-treated films, indicating relaxation of the stress that existed in the as-deposited films and change of its nature to tensile stress [81].

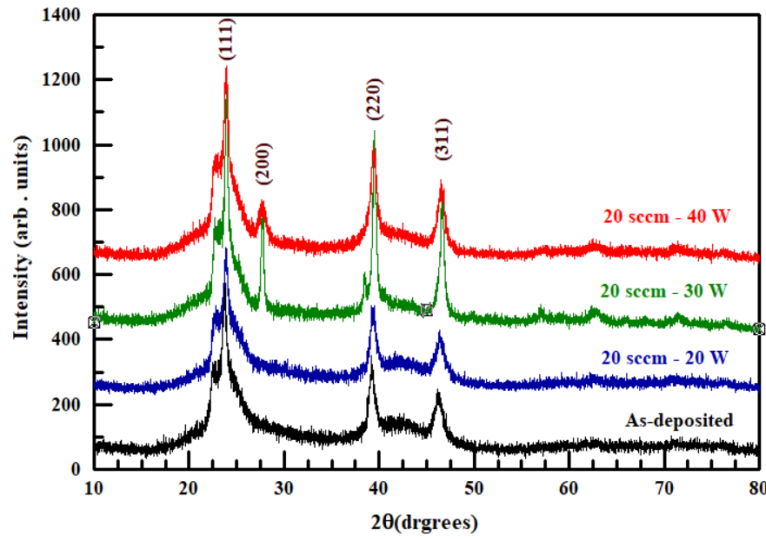


Figure 3.2 XRD patterns of the films before and after plasma treatments at a flow rate of 20 sccm and increasing powers.

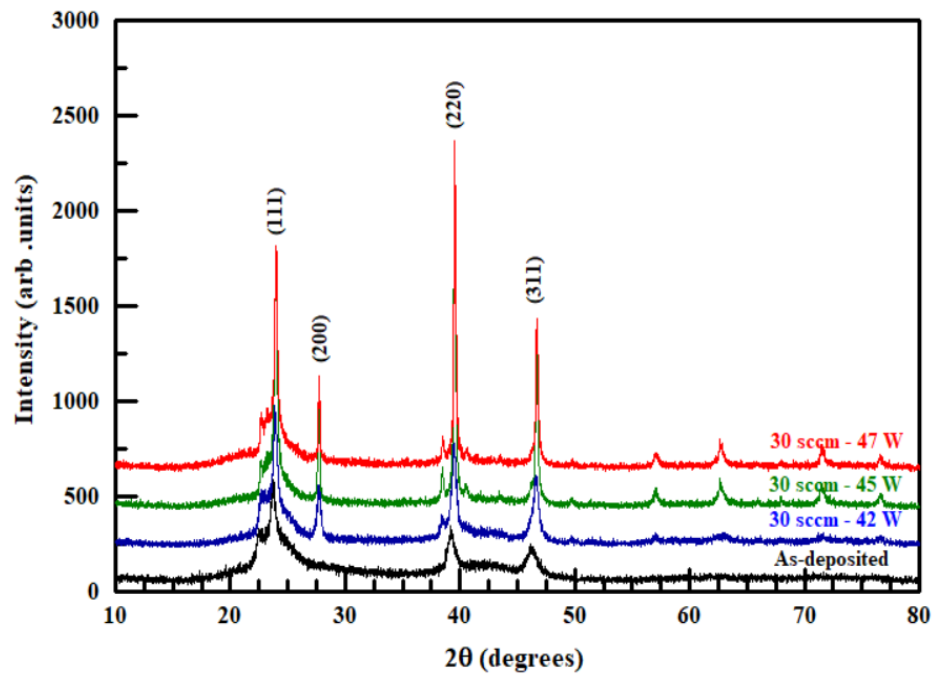


Figure 3.3. XRD patterns of the films before and after plasma treatments at a flow rate of 30 sccm and increasing powers.

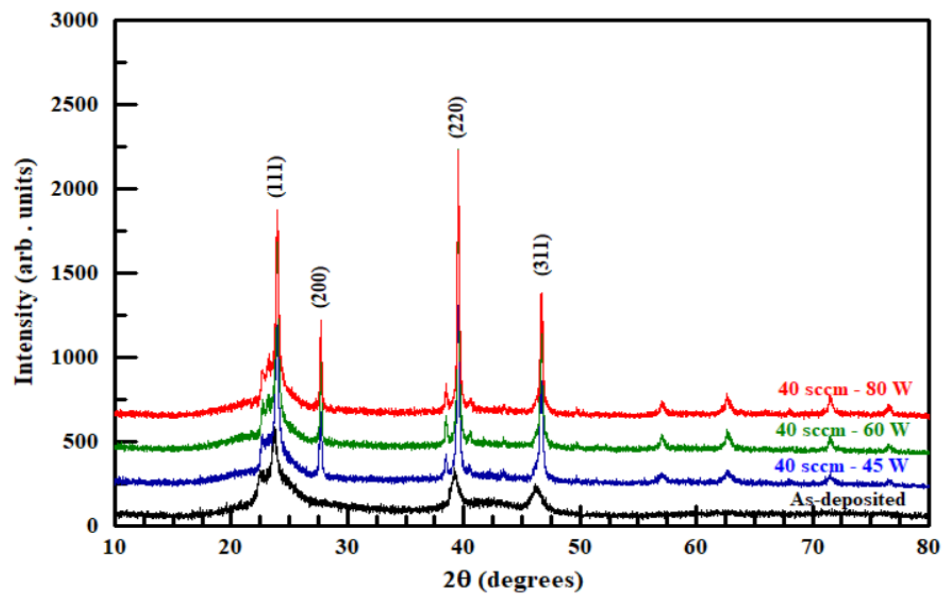


Figure 3.4 XRD patterns of the films before and after plasma treatments at a flow rate of 40 sccm and increasing powers.

Table 3.1. Structural parameters of the films

Ar flow rate (sccm)	<i>P</i> (W)	<i>d</i> (nm)	$(2\theta)_{111}$	<i>L</i> (nm)	<i>C</i> ₁₁₁	<i>C</i> ₂₂₀	<i>C</i> ₃₁₁	<i>C</i> ₂₀₀	<i>f</i>	<i>a</i> (Å)	$(d_f)_{111}$ (Å)	ε
.....		1654	23.71°	12.52	0	0.83	1.08		0.120	6.493	3.75	+0.175
20	20	1627	23.85°	11.42	1.10	0.87	1.02		0.095	6.455	3.73	-0.405
20	30	1305	23.93°	20.22	0.60	1.65	1.50	0.25	0.591	6.434	3.71	-0.737
20	40	1287	23.89°	12.52	1.26	1.12	1.41	0.21	0.467	6.446	3.72	-0.549
30	42	1319	23.89°	19.39	0.91	1.12	1.58	0.39	0.427	6.445	3.72	-0.561
30	45	1193	23.94°	30.23	0.61	1.53	1.60	0.26	0.579	6.433	3.71	-0.746
30	47	1099	23.96°	29.77	0.57	1.68	1.53	0.22	0.384	6.426	3.71	-0.851
40	45	1269	23.90°	25.72	0.63	1.41	1.67	0.29	0.561	6.442	3.72	-0.606
40	60	1074	23.93°	30.87	0.60	1.65	1.50	0.25	0.591	6.434	3.71	-0.738
40	80	1027	23.94°	29.92	0.66	1.54	1.54	0.25	0.562	6.433	3.71	-0.747

P: plasma power, *d*: film thickness, $(2\theta)_{111}$: the angular position of the XRD (111) peak, *L*: crystallite size,
*c*₁₁₁, *c*₂₂₀, *c*₃₁₁, *c*₂₀₀: texture coefficient *C*_{hkl} for crystalline growth along the (hkl) direction, *f*: orientation factor,
 $(d_f)_{111}$: lattice spacing of the films along the (111) direction, *a*: lattice constant, ε : strain,

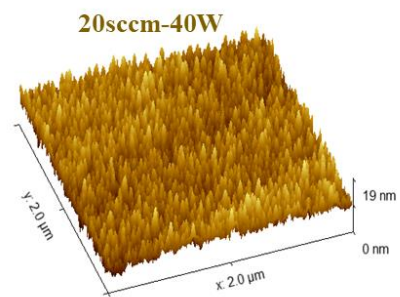
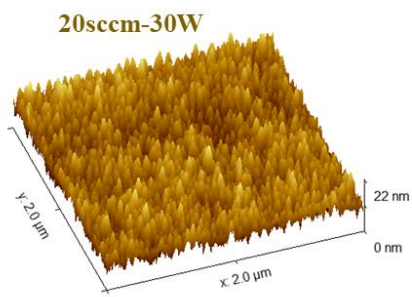
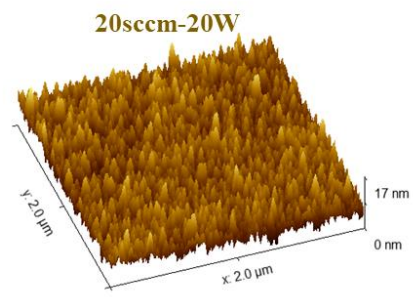
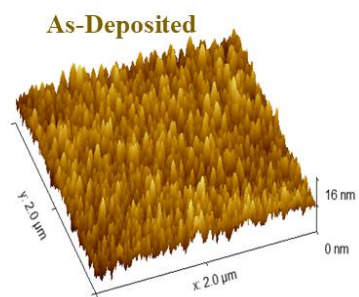
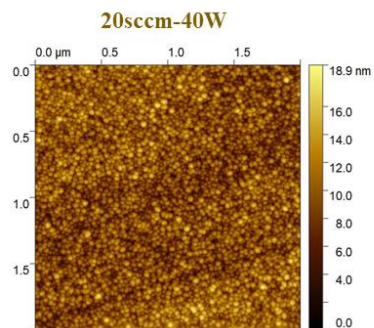
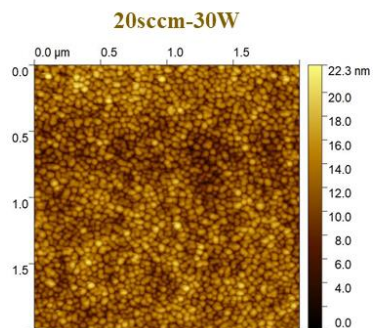
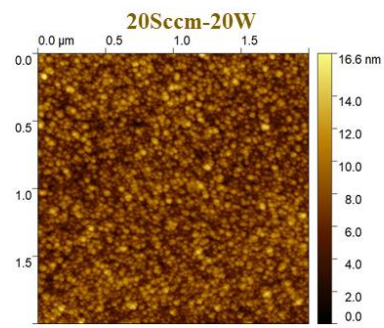
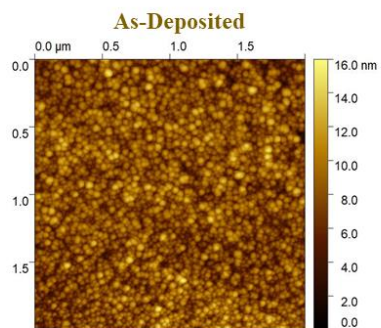
3.3 MORPHOLOGICAL ANALYSIS

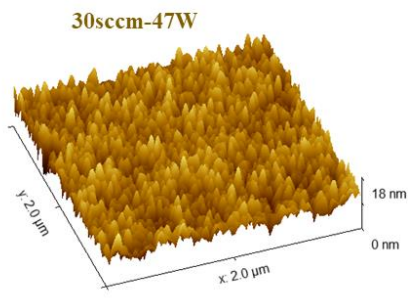
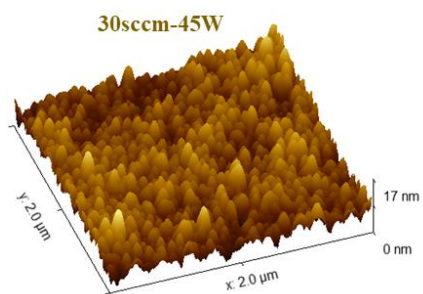
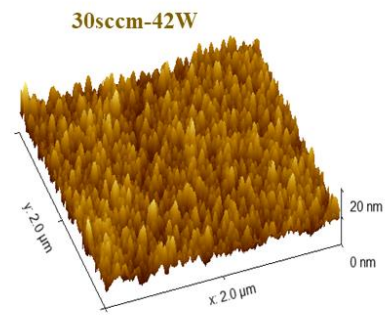
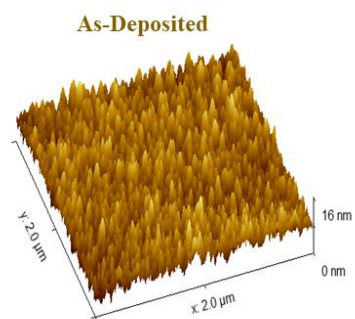
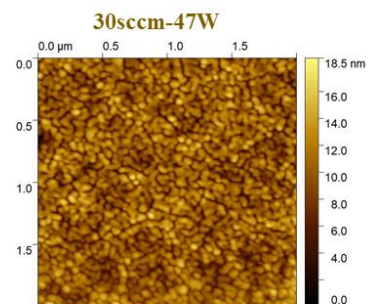
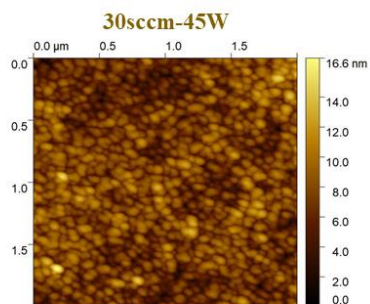
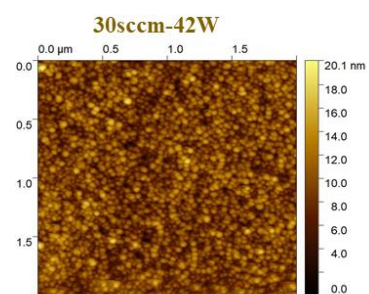
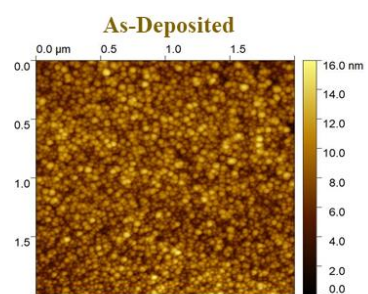
AFM was used to characterize the films' surface morphology and estimate the surface root-mean-square roughness (R_{rms}), which is a statistical measure used to quantify surface roughness. RMS roughness is calculated by squaring the height of each point on the surface (relative to a mean plane), averaging those squared values, and then taking the square root of that average. This method emphasizes larger deviations or rough spots on the surface, making it useful for assessing the overall surface texture. Figure (3.5) shows two-dimensional (2D) and three-dimensional (3D) AFM images of CdTe thin film surfaces before and after plasma treatment. The as-deposited films exhibited a homogeneous microstructure that was free of voids, pinholes, or microcracks. Statistical analysis of the AFM images revealed the root-mean-square surface roughness (R_{rms}), whose values are listed in Table (3.2). The as-deposited films had an R_{rms} value of 1.90 nm, which is less than 0.1% of the film's thickness, indicating a highly smooth surface, which is typical of thermal evaporation on unheated substrates. Plasma treatment resulted in a progressive increase in surface roughness that may be attributed to the etching effect caused by plasma treatment [50,84,85]. At higher plasma powers (60 W, and 80 W), the R_{rms} started to decrease to be less than the R_{rms} of the as-deposited films. Decrease in RMS surface roughness: indicates that the surface has become smoother. The peaks and valleys on the surface are less pronounced, and the overall texture is more uniform.

Table 3.2 Morphological analysis of the films

Ar flow rate (sccm)	<i>P</i> (W)	(<i>R_{rms}</i>) (nm)
.....		1.90
20	20	1.93
20	30	2.73
20	40	2.32
30	42	2.35
30	45	1.94
30	47	2.19
40	45	2.85
40	60	1.41
40	80	1.34

R_{rms}: root-mean-square surface roughness, *P*: plasma power





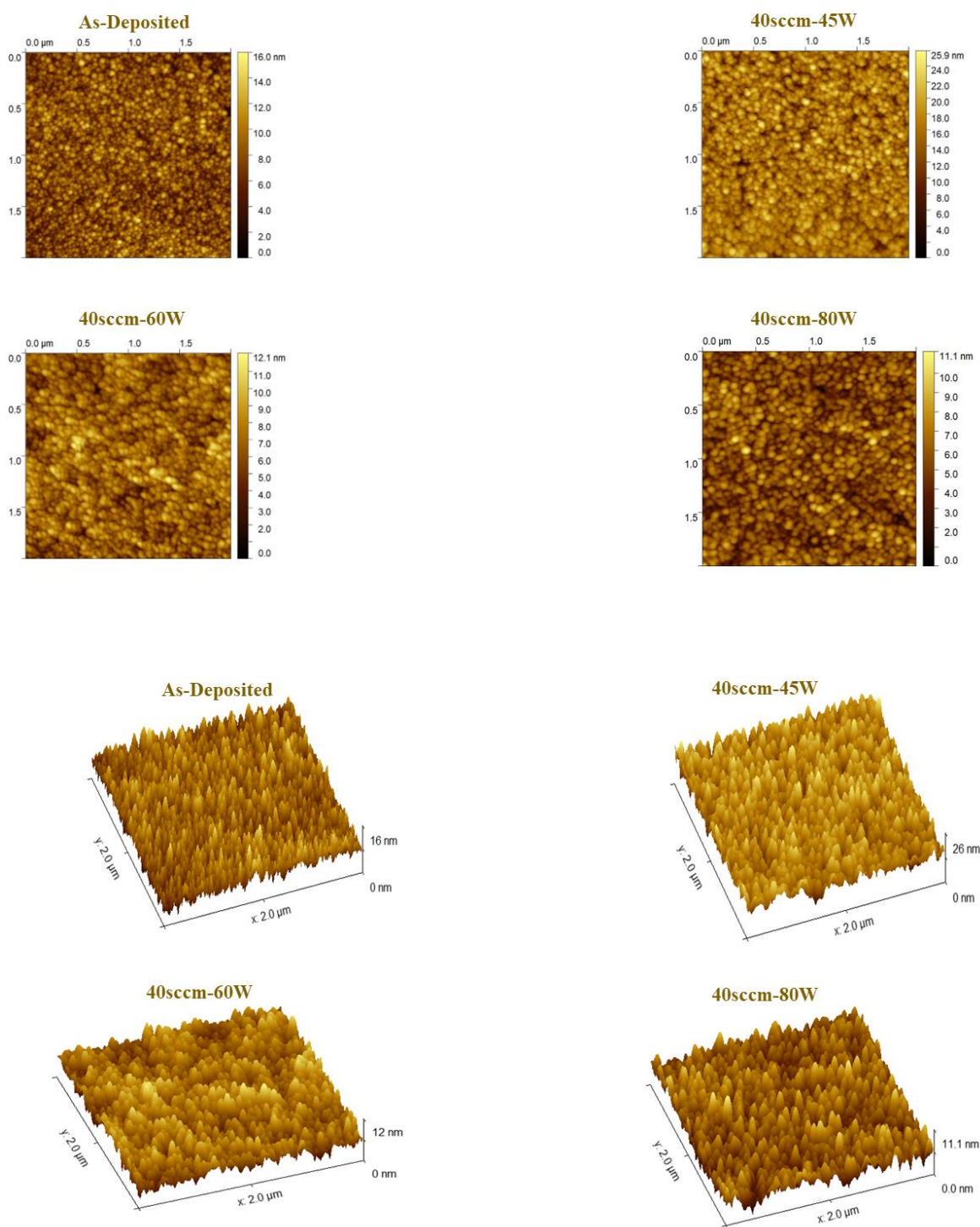


Figure 3.5 2D and 3D AFM images of CdTe thin film surfaces before and after plasma treatment.

CHAPTER 4

OPTOELECTRICAL PROPERTIES

4.1 OPTICAL PROPERTIES

The optical properties of thin films, such as the absorption coefficient (α) and the band gap (E_g), are significantly dependent on the preparation conditions and the techniques used to extract the optical constants from measured data. It is crucial to determine these properties as functions of light wavelength (or photon energy).

The optical spectra of the films prior to and after plasma treatment are illustrated in Figure (4.1). The existence of extrema (maxima and minima) can be attributed to the interference of numerous light beams reflected from the film's front and back surfaces [86,87]. This indicates the presence of distinct film/air and film/substrate interfaces, thereby signifying the superior optical quality of the films. The as-deposited film demonstrated significant transparency in the near-infrared spectrum, with a minor decrease observed following plasma treatment at $P = 20$ W. However, the transmittance was significantly diminished for the films exposed to $P \geq 30$ W. This may be ascribed to the increased light scattering resulting from higher surface roughness values [40]. In the fundamental absorption region, the absorption coefficient α is approximately related to the transmittance T as follows [88]:

$$\alpha = \left(\frac{1}{d}\right) \ln(1/T) \quad (4.1)$$

The values of α are shown in Figure (4.2). The as-deposited films had $\alpha \geq 10^4 \text{ cm}^{-1}$ in the (Near-Infrared region) NIR region, reflecting the superior suitability of CdTe as a

photovoltaic material. There was a slight increase in the absorption coefficient when the films were treated with $P = 20$ W. However, the films treated with $P \geq 30$ W exhibited a substantial increase in the absorption coefficient. This could be attributed to a complex interplay of the factors affecting absorption: polycrystallinity, surface roughness, and chemical defects. In the vicinity of the fundamental absorption region, the absorption coefficient is related to photon energy (E) by the Tauc relation [42,47]:

$$\alpha E = B(E - E_g)^\eta \quad (4.2)$$

where B is a constant, , and η is an exponent that depends on the bandgap type, where $\eta = 1/2$ or 2 for a direct or an indirect bandgap, respectively. A plot of $(\alpha E)^{1/\eta}$ versus photon energy shows a linear portion whose horizontal intercept yields the bandgap, as shown in Figures (4.3,4.4). The values of the direct and indirect band gaps are summarized in Table 4.1 as a function of plasma power. The direct bandgap was 1.440 ± 0.005 eV, unaffected by plasma treatment. Numerous previous reports have indicated the independence of the direct bandgap in plasma treatment [89,90]. However, the indirect bandgap was significantly reduced due to plasma treatment. This is expected given the dependence of the indirect bandgap on the density of structural and chemical defects that induce energy levels within the forbidden bandgap, resulting in bandgap reduction. The same effect was previously seen in the absorption coefficient.

Table 4.1 band gap values

Power	Direct Band Gap	Indirect Band Gap
(W)	(eV)	(eV)
0	1.45	1.13
20	1.44	1.09
30	1.44	0.92
40	1.44	0.88
42	1.46	0.86
45	1.47	0.87
47	1.47	0.85
45	1.46	0.86
60	1.48	0.85
80	1.47	0.92

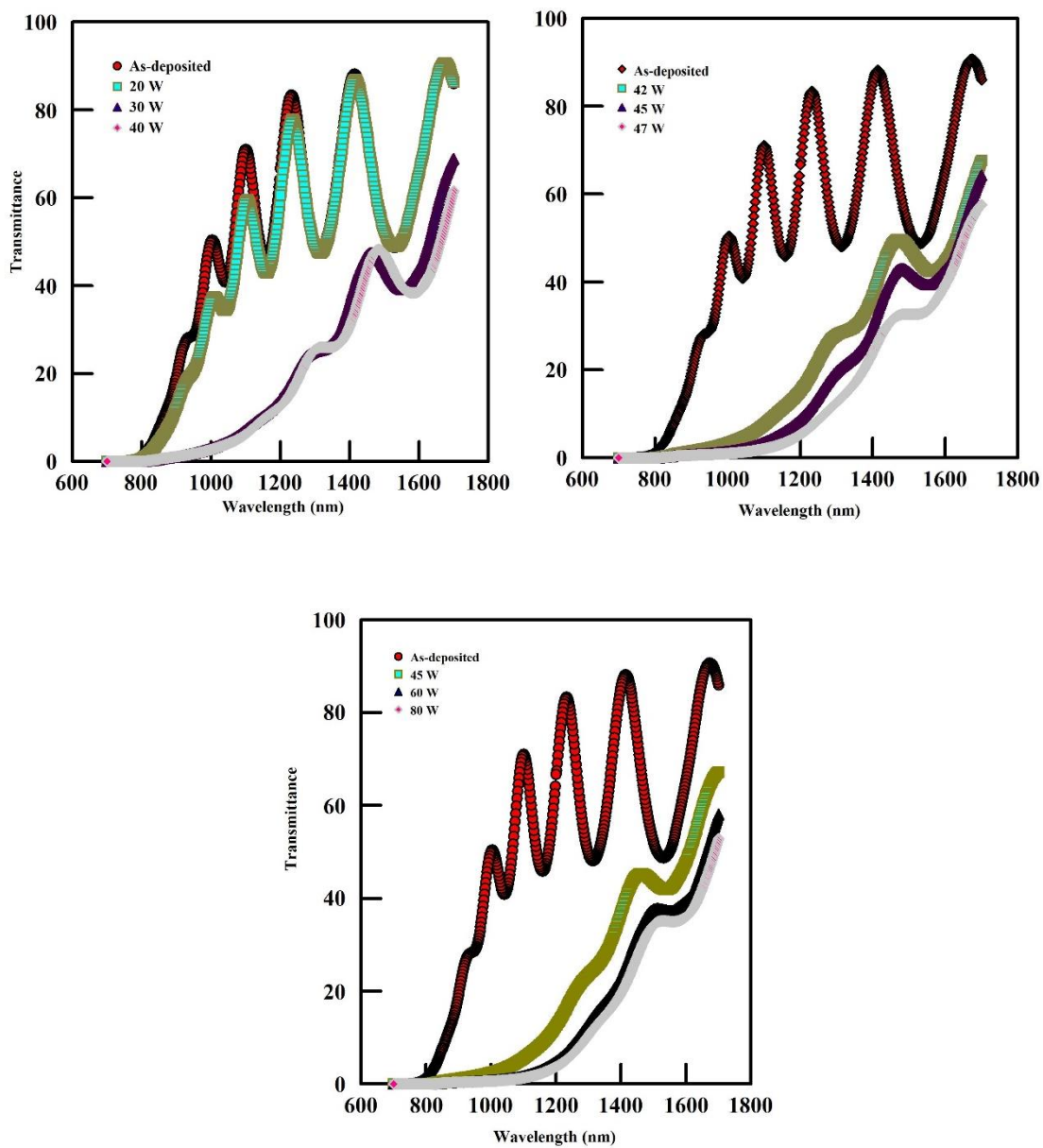


Figure 4.1 The normal-incidence transmittance spectra of the CdTe films as functions of plasma power.

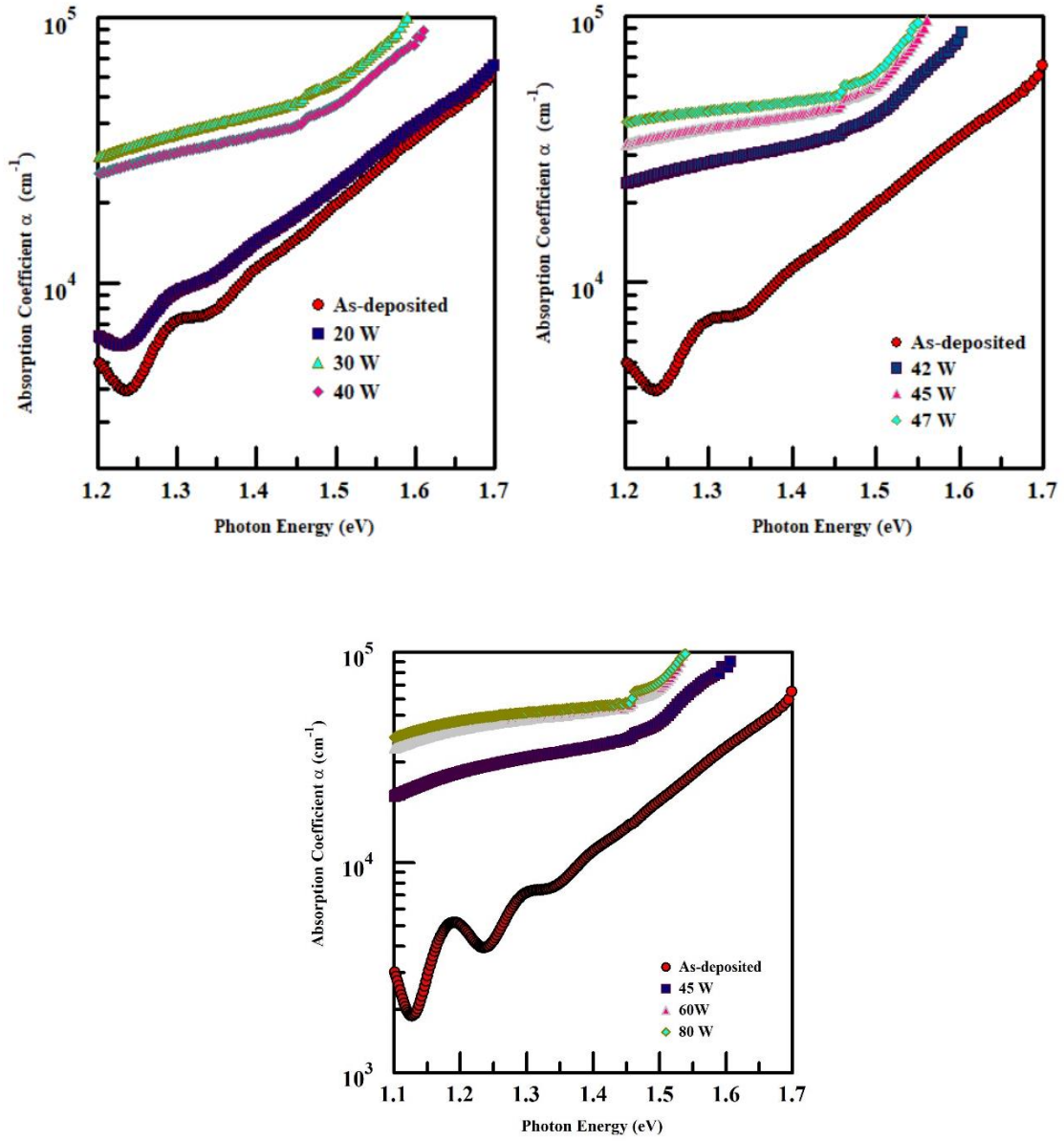


Figure 4.2 Absorption coefficients of the CdTe thin films as functions of plasma power.

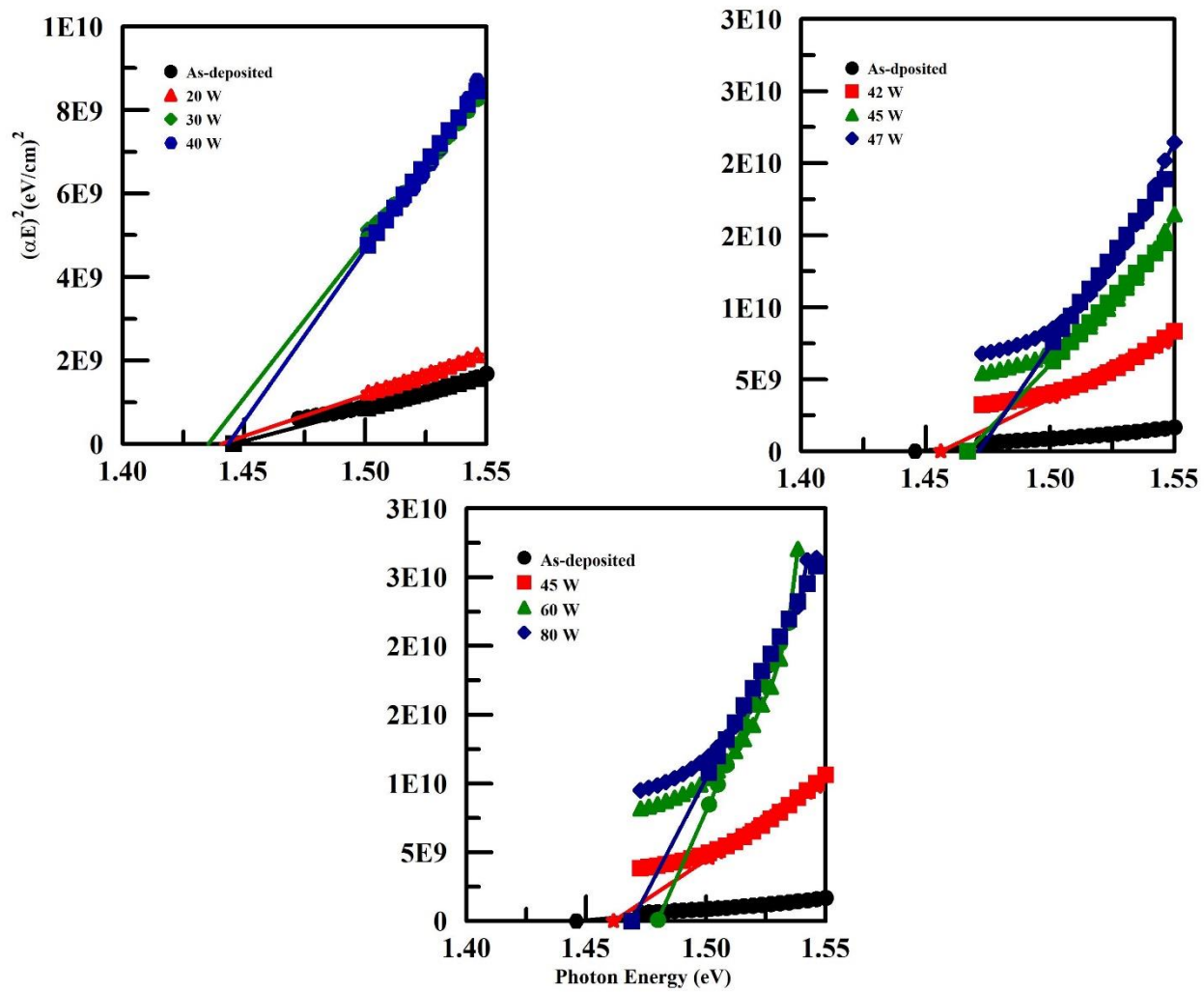


Figure 4.3 Tauc plot for extracting the direct bandgaps of the CdTe thin films as functions of plasma power

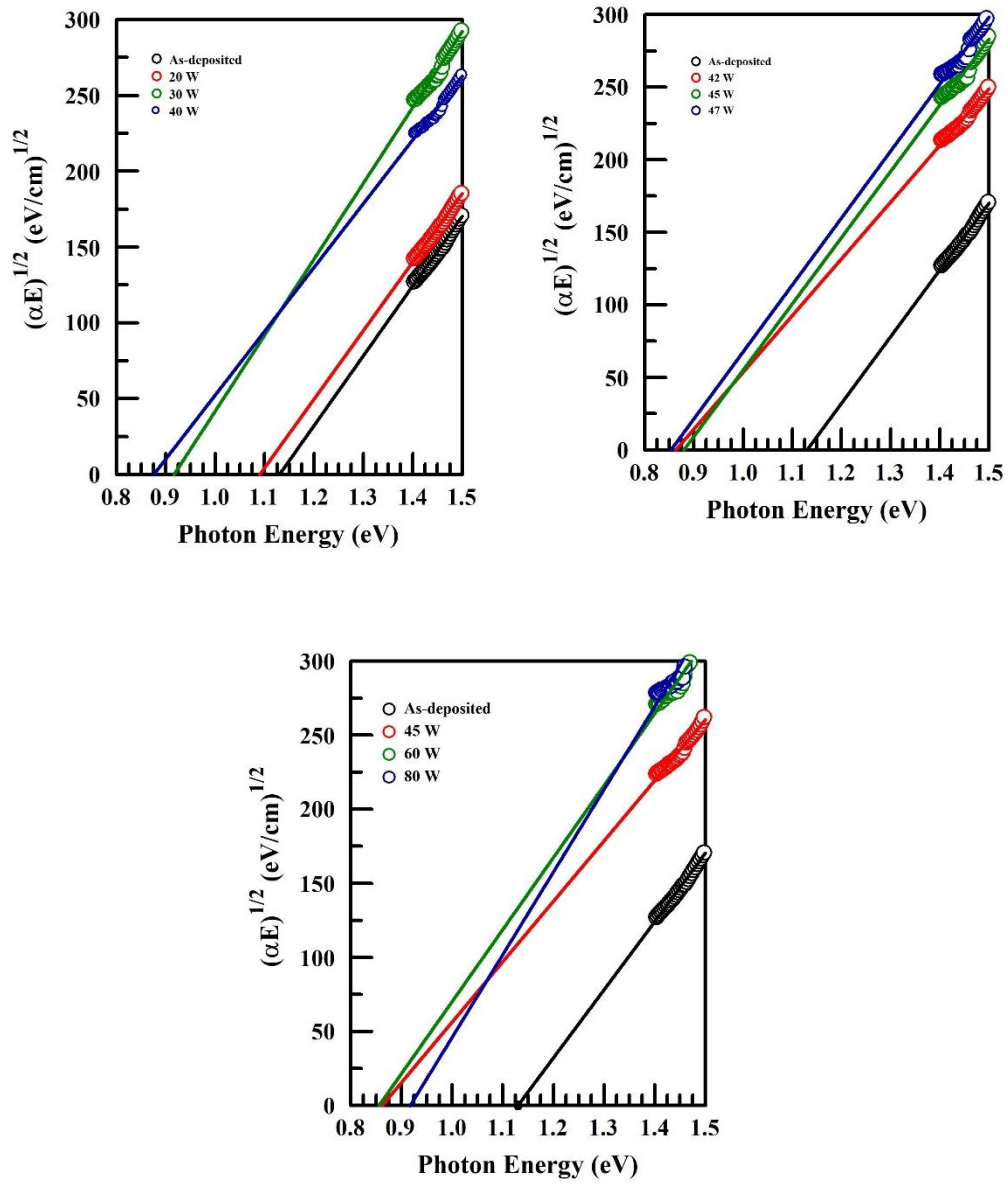


Figure 4.4 Tauc plot for extracting the indirect bandgaps of the CdTe thin films as functions of plasma power

4.2 ELECTRICAL PROPERTIES

Plasma treatment of semiconductor thin films is known to enhance their electrical conductivity [40,43,47,51,52,84], and the enhancement mechanism depends on the type of material involved (such as oxides, tellurides, or selenides), along with the nature of the defects and the donor/acceptor levels. The current-voltage curves of the films are obtained in Figure (4.5). For the as-deposited film, the dark current was in the range of 10^{-9} A, which is very close to the low-power plasma-treated film (20 W). Upon treatment by plasma at higher power (30 W), the dark current increased to be in the range of 10^{-5} A, and for a higher plasma power of 40 W at the same argon flow rate, the dark current became in the range of 10^{-6} A which is still higher than the as-deposited one. For higher argon flow rate and higher plasma power, the dark current decreased to be in the range of 10^{-10} A, which is less than the as-deposited one. For all films, the photocurrent is double the dark current, which is not a big difference because the increment in free carrier concentration due to photo-generated electron-hole pairs was insignificant compared to the large background free electron concentration in the films. The $I-V$ curves were used to calculate the dark resistivity (ρ) of the films using a two-probe technique using the relation [91]:

$$\rho = \frac{RwA}{l} \quad (4.3)$$

where w is the length of the gold electrodes (2.54 cm), l is the separation between the two electrodes (2 mm), and R is the resistance of the film calculated as the inverse of the slope of the $I-V$ curve. The values for the resistivity are listed in Table (4.2). The resistivity of the as-deposited films was $3.9 \times 10^4 \Omega \cdot \text{cm}$. When treated with a plasma power of 20 W, the resistivity increased to $1.1 \times 10^5 \Omega \cdot \text{cm}$, possibly due to a reduction in crystallite size.

Conversely, plasma treatment with a power of 30 W decreased resistivity by two orders of magnitude to $1.9 \times 10^2 \Omega \cdot \text{cm}$, representing the films with the lowest resistivity in the study. This substantial decrease is attributed to a combination of factors. These films exhibited the largest crystallite size and orientation factor, contributing to improved charge carrier mobility and enhanced electrical conductivity [92]. Ultimately, when plasma power was increased to 40 W, the resistivity increased to $1.1 \times 10^3 \Omega \cdot \text{cm}$, although it was lower than the pristine films. For higher plasma powers and higher argon flow rates, the resistivity increased to be in the range of 10^5 and 10^6 , which coincides with the photo and dark current values. the ρ variation closely follows the degree of crystalline perfection of the films, which can be defined as $\phi \equiv LC_{111}$. the least value of ϕ has the least resistivity value. Better assessment of the influence of plasma treatment would be accomplished by comparing the differences in the currents caused by plasma treatment. A plasma photoresponse G may be defined as follows:

$$G = \frac{I_{light} - I_{dark}}{I_{dark}} \quad (4.4)$$

where I_{dark} is the dark current of the film I_{light} is the corresponding photocurrent, The values of G are shown in Figure (4.6). The as-deposited films had a mean photoresponse of 34.5, exhibiting the distinct suitability of CdTe as a photovoltaic material. When treated with a plasma power of 20 W, the mean sensitivity decreased to 9.8, which qualifies these films for photovoltaic applications. The reduction in sensitivity could be attributed to the lower crystallinity of the plasma-treated films. The films treated with higher plasma powers ($P \geq 30$ W) showed a significant reduction in sensitivity to mean values of 1.3 and 1.6 for $P = 30$ W and 40 W, respectively. The films treated with plasma power 47 W showed a

blow-up in photoresponse to a mean value of 74.1 due to the large direct current. (The reduction in sensitivity for the other films is attributed to their larger intrinsic dark current. In this case, the photocurrent represents a minor increase in current, hence the reduced sensitivity.

Ar flow rate (sccm)	Power (W)	Resistivity ($\Omega\cdot\text{cm}$)	Φ (nm)
.....		3.9×10^4	136.468
20	20	1.1×10^5	125.62
20	30	1.9×10^2	121.32
20	40	1.1×10^3	157.752
30	42	1.7×10^6	176.449
30	45	1.9×10^6	184.403
30	47	1.1×10^6	169.689
40	45	1.3×10^6	162.036
40	60	7.3×10^5	185.22
40	80	7.1×10^5	197.472

Table 4.2 shows the electrical parameters of the films

Φ :crystalline perfection

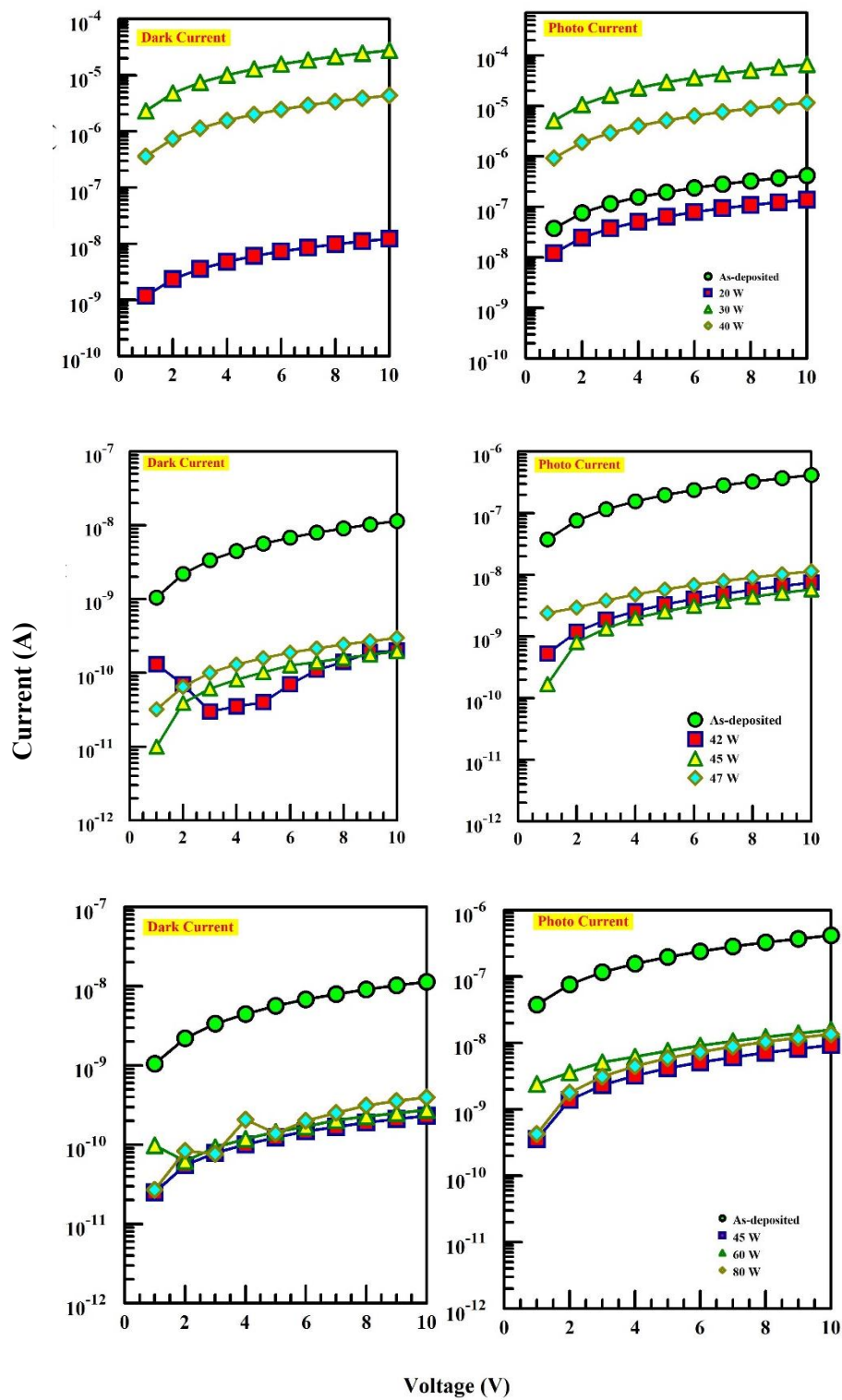


Figure 4.5 Current-voltage curves of the CdTe thin films as functions of plasma power: dark current (left) and photocurrent (right).

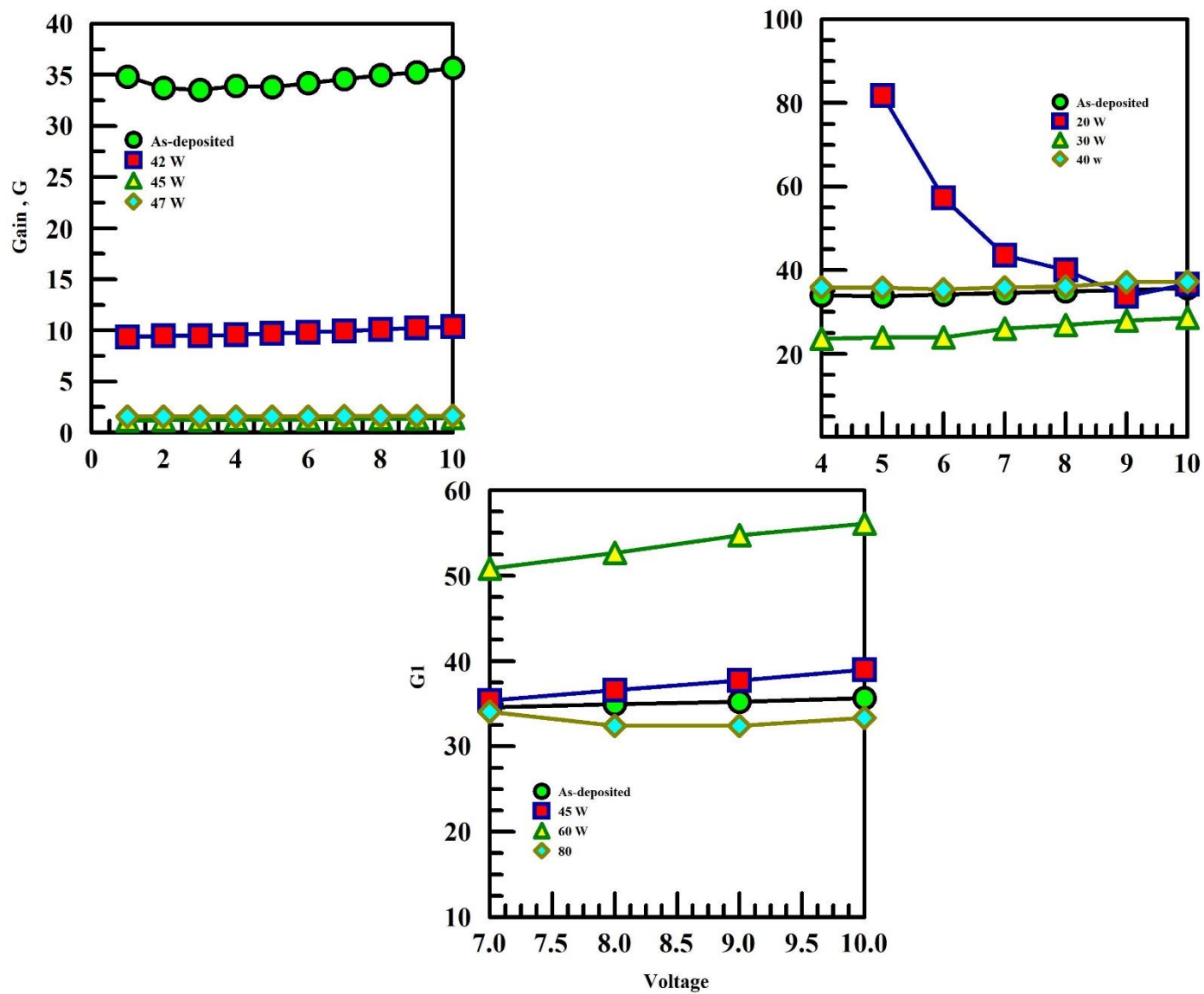


Figure 4.6 photoresponse of CdTe thin films as a function of voltage and plasma power.

CHAPTER 5

CHEMICAL PROPERTIES

The chemical properties are studied by using two techniques, XPS and SIMS.

5.1 XPS ANALYSIS

XPS was used to investigate the influence of plasma treatment on surface chemistry. Due to spin-orbit splitting, cadmium and tellurium have $3d$ core levels that consist of two sublevels ($3d_{3/2}$ and $3d_{5/2}$). In CdTe, the binding energies (BE) of Cd $3d_{5/2}$ and Cd $d_{3/2}$ are in the ranges 404.8–405.5 eV and 411.9–412.0 eV, respectively, amounting to a mean spin-orbit splitting (δ) of 6.7 eV [86]. The corresponding values for tellurium are BE ($3d_{5/2}$) = 572.3–572.9 eV, BE ($3d_{3/2}$) = 582.9–583.0 eV, and δ = 10.39 eV [86]. The structural, optical, and electrical properties of the samples have revealed that the optimum argon flow rate is 20 sccm and the optimum plasma power is 30 W. Therefore, the chemical properties of the samples will be studied with an argon flow rate of 20 sccm and different plasma powers. In Figure (5.1), the XPS spectra for the chosen samples are displayed in the core-level regions of Cd $3d$ and Te $3d$, and Table (5.1) lists the values of BE, δ , and the Te/Cd ratio. Our values are well within the reported ranges for BE and δ . The values clearly show that plasma treatment significantly impacted the chemical composition of the film surfaces. The Cd/Te ratio of the pristine films shows that the surfaces of these films were tellurium-rich. This ratio decreased as the films were treated with a plasma power of 20 W, indicating preferential sputtering of tellurium for this power. The ratio dropped even more, approaching the stoichiometric value of 1:1 when the plasma power was set to 30 W.

Nevertheless, the ratio increased significantly when the samples were treated with a plasma power of 40 W, indicating that preferential sputtering is a function of plasma power. Moreover, XPS analysis indicated selective oxidation of tellurium on the surfaces of the films treated with plasma power $P \geq 30$ W. The BE of the oxide component matches that of TeO_2 [86]. This oxidation is selective regarding the element affected and the plasma power. The source of oxygen could be residual oxygen in the Ar gas and the oxygen adsorbed on the surface during the transfer from the deposition chamber to the plasma system. The increase in plasma power led to significant oxidation, as evidenced by the increase in the ratio (R) of the oxide component to the total Te 3*d* spectrum, as calculated from the areas under the curves.

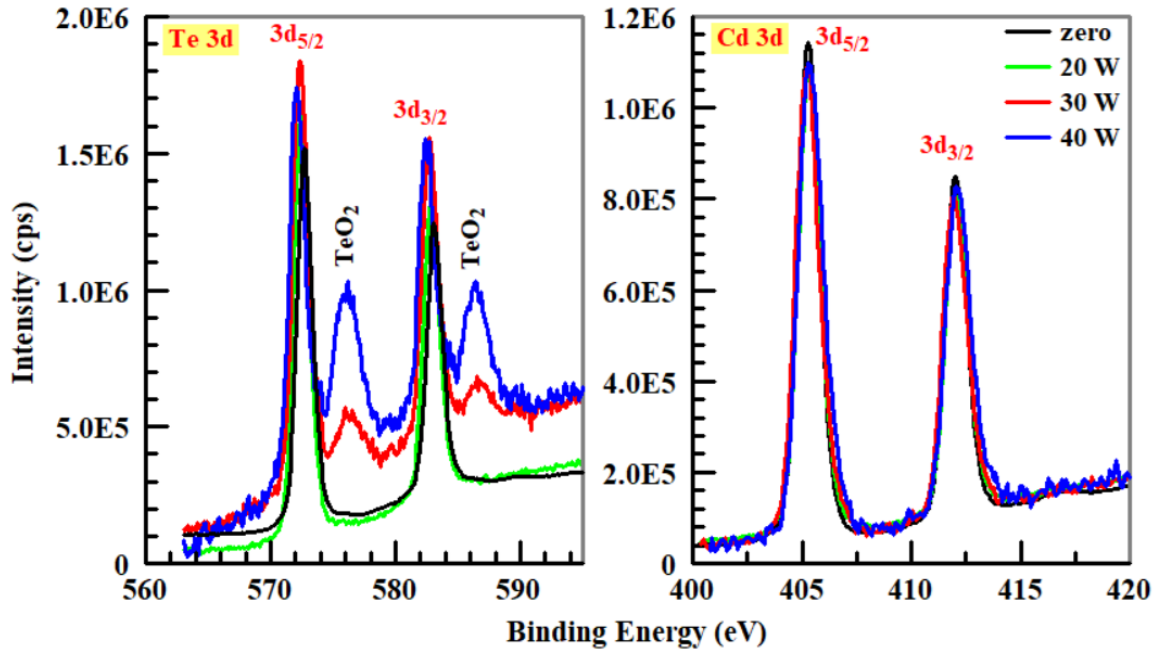


Figure 5.1 XPS spectra of the films in the Cd 3d and Te 3d core level regions.

Table 5.1. Chemical properties of the films

P	Cd 3d			Te 3d				Te/Cd	
(W)	BE (eV)		δ	BE (eV) in		BE (eV) in		δ	R
			(eV)	CdTe		TeO ₂		(eV)	
	$3d_{5/2}$	$3d_{3/2}$		$3d_{5/2}$	$3d_{3/2}$	$3d_{5/2}$	$3d_{3/2}$		
0	405.4	412.1	6.7	572.8	583.2	---	---	10.4	---
20	405.3	412.1	6.8	572.8	583.1	---	---	10.3	---
30	405.2	411.9	6.7	572.5	582.9	576.4	586.7	10.4	0.19
40	405.2	411.9	6.7	572.6	583.0	576.6	586.7	10.4	0.45

BE: binding energy, δ : spin-orbit splitting, R : ratio of TeO₂/CdTe

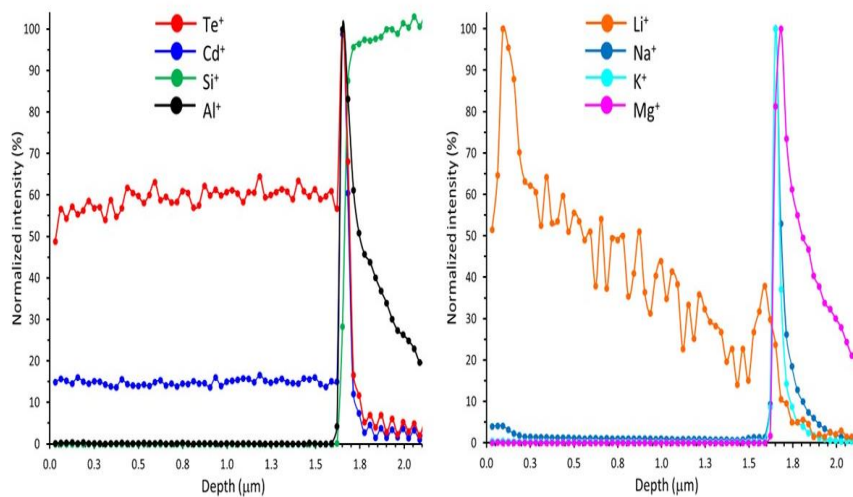
5.2 TOF-SIMS:

The samples were analyzed with TOF-SIMS depth profiling analysis. The analyses were only performed in positive ion. To efficiently compare the resulting depth profiles, the depth profile intensities have all been normalized to 100%, which makes it easier to perform comparisons. TOF-SIMS is not directly quantitative, but it is semi-quantitative under certain conditions. One ion intensity from one particular compound can, therefore, be compared with an identical ion intensity if the chemical surroundings are the same (like in this case, where all films consist of CdTe). The in-depth intensity variation of a particular ion is, therefore, semi-quantitative.

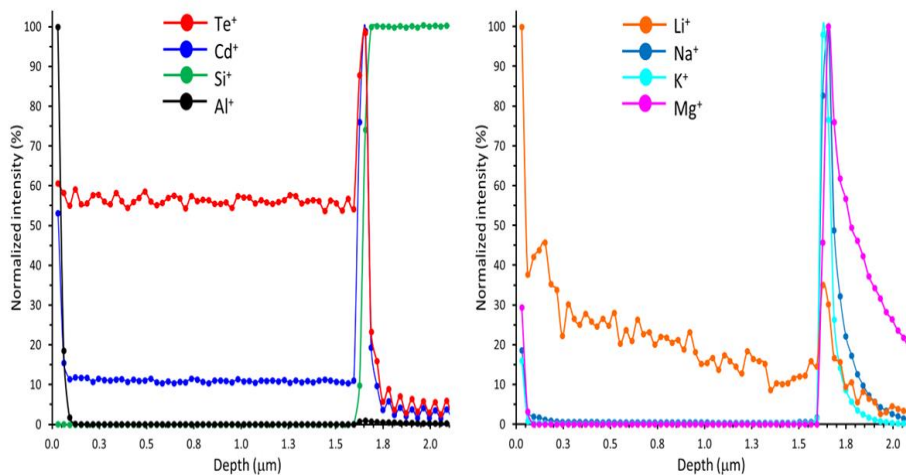
It has been found that the reference sample (As-deposited) consists mainly of Cd and Te with a trace amount of Li, Na, Al, and K (the Li⁺ signal is noisy, indicating a very low intensity/concentration). Figure (5.2) shows profiles at an Ar flow rate of 20 Sccm with increasing plasma power 20, 30, and 40 W. At 20 W, we are observing a beginning of surface segregation of Na, K, Mg, and especially Al. Going from 20 W to 30 W has a drastic effect that doesn't change much when continuing to 40 W. At 30 W and 40 W power, there is no significant surface segregation. Metals don't have a vapor pressure at these temperatures, so the species accumulating on the surface must come from the sample itself. The metals segregating to the surface are all typical glass components, so it is obvious to assume that they are segregated from the glass to the CdTe film surface. This is a big surprise that plasma can induce surface segregation from the substrate. That means at 30 W and 40 W, it is no longer a surface modification but a modification of the bulk close to

the surface that is modified, which will significantly change the physical properties of the material, which illustrates the behavior of the films.

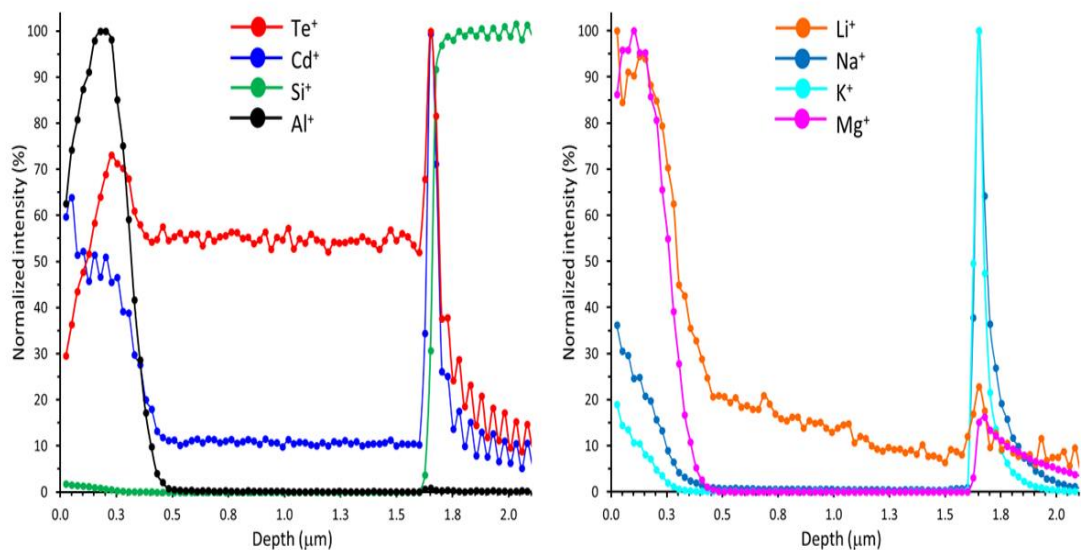
As-deposited (Reference sample)



Ar flow = 20 Sccm, 20W



Ar flow = 20 Sccm, 30W



Ar flow = 20 Sccm, 40W

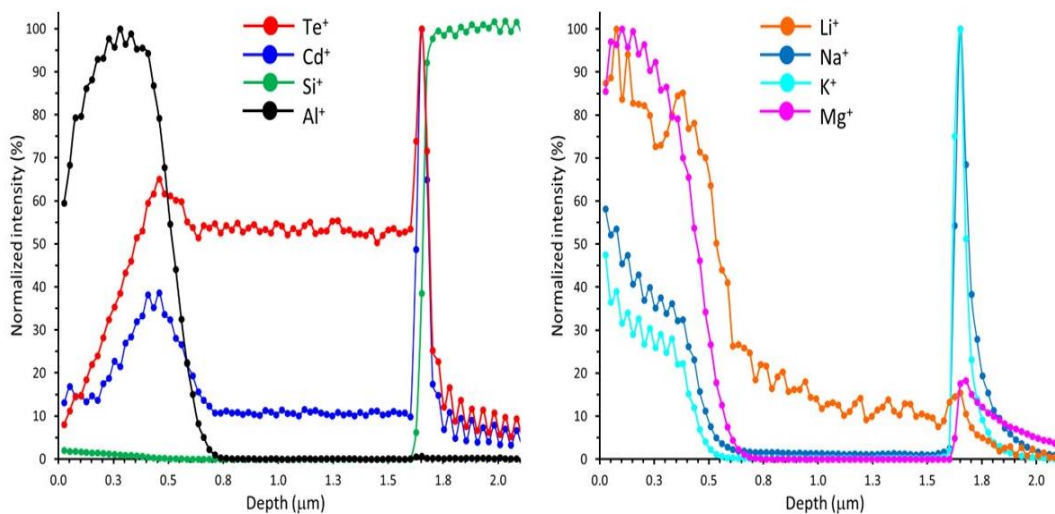


Figure 5.2 The SIMS profiles of the films before and after plasma treatment

CHAPTER 6

CONCLUSIONS AND FUTURE SUGGESTIONS

6.1 CONCLUSIONS

In the present work, CdTe thin films were fabricated by thermal evaporation of CdTe powder. The effect of post-deposition plasma treatment was investigated. The films underwent argon plasma irradiation for 1 hour. While the argon flow rate varied between 20, 30 and 40 sccm, the plasma power was varied in the range from 20 W to 80 W.

From the work, the following conclusions can be made.

- The thermal evaporation of CdTe resulted in films with a thickness of 1654 nm.
- Due to plasma etching, the thickness decreased. At a power $P = 20$ W, there was a negligible thickness reduction. At higher powers $P \geq 30$ W, a decrease in thickness occurred due to the plasma etching.
- The films had a polycrystalline structure with nano-sized crystallites (peaking at 20.22 nm when exposed to 30 W plasma power).
- As the power of the plasma was increased to 30 W, its greater thermal energy coalesced smaller grains to form larger grains.
- The crystallite size and orientation factor decreased as the power increased to 40 W. Then, the crystallite size started to increase at higher powers, which means that thermal energy is sufficient to overcome the defect density generation by the plasma.

- AFM imaging revealed the surface morphology of the films as uniform, void, pinhole, and microcrack-free microstructures.
- The influence of plasma treatment on surface chemistry was investigated using XPS. The results show that the plasma treatment significantly impacted the chemical composition of the film surfaces.
- Furthermore, the tellurium-to-cadmium ratio varied as the plasma power varied.
- The depth profiling investigated by TOF-SIMS measurements indicated that plasma may induce surface segregation from the substrate.
- At 30 W and 40 W power, there is no significant surface segregation. Metals don't have a vapor pressure at these temperatures, so the species accumulating on the surface must come from the sample itself. The metals segregating to the surface are all typical glass components, so it is obvious to assume that they are segregated from the glass to the CdTe film surface.
- High transparency was observed in the near-infrared range, reducing slightly after a plasma treatment at $P = 20$ W. The transmittance was drastically reduced for the films irradiated with $P \geq 30$ W.
- However, the direct bandgap (1.44 eV) remained unaffected by the plasma treatment.
- The indirect band gap was affected by plasma.
- The electrical resistivity of the initially deposited films was $3.9 \times 10^4 \Omega \cdot \text{cm}$, emphasizing the resistive nature of the as-deposited CdTe.
- The resistivity was oscillated with changes in the plasma power. Films treated with 30 W plasma power displayed the lowest resistivity at $1.9 \times 10^2 \Omega \cdot \text{cm}$.

- According to the current-voltage measurements, the electric current increased under light illumination, an expected characteristic of an efficient photovoltaic material.
- At 30 W and 40 W, the current increased to the range of μA compared to the nA, for the as-deposited films corresponding to the same voltage.

In conclusion, optimal performance was achieved with a 30 W plasma treatment, yielding films at 80% of the original thickness. While the bandgap remained unaffected, optical absorption underwent significant development. With their larger crystallite size and surface roughness, these films possess relatively high electrical conductivity, leading to an amplified photoresponse.

6.2 SUGGESTIONS

In the future,

- CdTe thin film can be prepared by e-beam and compared with other techniques.
- Investigating the effect of post-deposition treatment of H₂ and O₂ plasma as a mixture with Ar gas.
- The exposure time to the plasma can be changed from 10 minutes to 1 hour, with time taken as another parameter.
- Instead of fused silica, CdTe can be deposited on crystalline silicon, and the effect of substrate type on the properties of the films can be studied.

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Publications

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