Journal of Radiation and Nuclear Applications
An International Journal

http://dx.doi.org/10.18576/jrna/060303

The Effect of Neutron Irradiation on Optical, Structural and Morphological Properties of Cadmium Selenide Thin Films

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Received: 7 Jun .2021, Revised: 22 Jul .2021, Accepted: 18 Aug. 2021.

Abstract: CdSe films were prepared using the chemical bath deposition method on glass bases and at temperatures of 50°C and 70°C, PH = 9, the CdSe films were exposed to neutron irradiation from the (Am^{241} – Be^{10}) source with a flux of 3×10^5 n/cm^2.s and energy 5.71MeV for a period of irradiation for a 7 days, the optical properties of irradiation were studied using UV - V spectrophotometer, and from the following measurements, XRD, FESEM, EDX, the shape and structure of the prepared films were determined. It was found that there is an effect of neutron irradiation on the optical and structural properties of CdSe films where it was found that there is a transform in the structure of films at a temperature of 70°C from the Cubic structure to hexagonal, and the energy gap increases after irradiation and the absorption coefficient (α) increases with irradiation due to an increase in the energy gap, and the particle size also increases with irradiation, while the relative density decreases with irradiation. The surface shape of the films is significantly affected when the films are irradiated with neutrons.

Keywords: Cadmium Selenide, Neutron flux, XRD.

1 Introduction

The II-VI semiconductor compounds belonging to the family of cadmium chalcogenide (CdS, CdSe, CdTe) are very important materials for photovoltaic applications[1]. CdSe thin films have a wide and direct energy gap (1.74 eV), n-type semiconductor nature [2], the interest that CdSe received is due to its importance in solar cell applications [3].

CdSe thin films were prepared using a variety of chemical and physical methods such as plating, CVD chemical deposition, sol-gel, chemical spray pyrolysis, thermal evaporation, laser ablation, electron beam. all of these methods require developed and expensive tools. Among all the thin film deposition methods, CBD chemical bath deposition is the simplest method that provides a great scope for manufacturing a large area [4,5], has a low cost and does not require expensive tools and a low deposition temperature, and this technique depends on the controlled release of metal ions [6] The rate of deposition can be controlled by adjusting the following parameters such as the bath temperature, the pH of the solution, the deposition time, and the concentration of the reactants [7]. They can be crystallized in hexagonal [8], cubic [9], or mixed (hexagonal – cubic)[10].

These films have applications in many semiconductor devices such as thin film transistors, solar cells, lasers, gas sensors, and photovoltaic [11]. Several studies have been investigated the effect of radiation on the properties of (CdSe) films such as Gamma and ^{60}Co. However, this study will address the effect of irradiation with neutron irradiation on CdSe properties.

The neutron is considered to be one of the particles with equal charge and the most portability on penetrable and It is followed by gamma rays [12], because of its equal charge, it does not interact with a matter with the Coulomb force, but can penetrate electrons without interaction [13].

When a neutron beam falls on a specific material, its intensity decreases as a result of the collision of neutrons with the nuclei of the film materials, this is done either by scattering (elastic and inelastic) or it can be captured by nuclei or materials with the emission of a gamma-ray [14,15]. Neutron irradiation causes damage to the film due to elastic collision in which the energy of the incident neutron is transferred to the primary knock-on atom (PKA), which contains a large part of the energies on the order of tens of KeV, allowing them to produce defect cascades that lead to the effective formation of interstitial-vacancy pairs of Frenkel defects [16] and As well as the events of atomic displacement and mechanical film stress [17].

This study aims to investigate the effect of neutron irradiation on CdSe thin films from where optical, structural properties, and the morphology surface.

2 Experimental Details

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2.1 CdSe Thin Film Preparation

A glass slides of dimensions (7.5×1.3×0.1) cm were used as the substrate, washed with running water and one of the cleaning powders, the clean slide placed in a solution of hydrochloric acid for 15min, then put in deionized water, immerse it in a hot alcohol solution, then in acetone, and then it is kept in a vacuum place to keep it from the air and the factors affecting it. The cadmium selenide films were prepared by chemical bath deposition technique(CBD) using Na_2SeSO_3 as a source of Se ions and $CdCl_2$ as a source of Cd ions. The selenium was mixed with Sodium Sulfite in 10ml of deionized water in the Reflux system and heated in a magnetic stirrer for two hours, the $CdCl_2$ was dissolved in 10ml of deionized water with the addition of ammonia NH_3, white turbidity appears which was dissolved more than ammonia solution and then a few drops added from Triethanolamine TEA. The slides were placed inside the solution vertically at 50°C for 3 hours to complete the deposition. This process was also repeated but at 70°C temperature.

2.2 Experimental Techniques

The samples were irradiated use neutron for 7 days, The neutron source produces fast neutrons. It is surrounded by paraffin, Working on attenuation of the fast neutron into thermal neutrons, due to its small mass number and high cross-sectional area. The samples were placed after paraffin wax at a distance of 4 cm from the neutron source [18]. When the neutrons pass inside the film, several collisions can lead to the deviation of the neutrons from their direction, in each collision they lose part of their energy and move away from the source of their emission and their intensity decreases in the falling beam according to the following law [19].

$I = I_\circ e^{\sigma n x}$

where $I_\circ$ is the intensity of the initial neutrons before they enter the material, σ is the total cross-section that represents the absorption and scattering of neutrons by the material it passes through, n is the number of atoms in a unit volume of a substance, x thickness of the distance traveled by the neutron inside the material.

The optical properties of CdSe films were measured using (UV-VIS-NIR) spectrophotometer. The transmittance and absorption were measured as a function of the wavelength of the range from (340-1000)nm and the optical absorption coefficient was calculated from the relationship: $\alpha = 2.303\ A/t$, where A: the absorption coefficient, and t: the thickness of the film.

The relationship between the square of the absorption coefficient and the energy of the incident photon was plotted to calculate the energy gap (E_g) of the film. The extinction coefficient represents the amount of energy absorbed in the thin film or the inertia that occurs in the electromagnetic wave inside the film and can be found from the following relationship [20]; $K = \frac{\alpha\lambda}{4\pi}$.

The Urbach energy represents the number of levels within the optical energy gap, which is indicative of the existence of different types of random levels and defects that arise according to the method used in preparing the film. The relationship between the photon energy and the absorption coefficient is given according to the following equation[21], $\alpha = \alpha_\circ \exp(\frac{h\nu}{E_U})$.

An XRD device was also used to identify the crystal structure of pure and irradiated films by comparing it with the standard card for X-ray diffraction (JCPDS).

2.3 The Interaction of a Neutron with Matter

Neutrons interact in several ways, depending on the energy of the neutron and the type of material reactants, divided into several sections, including:

1-Scattering reactions (elastic scattering and inelastic scattering): When a neutron interacts with the nucleus of an atom and the target nucleus remains in the same state after the interaction, and part of the kinetic energy is transferred to the nucleus during the collision, this interaction is subject to the laws of conservation of energy and momentum. This state is known as elastic scattering [22], But when the incident neutron is absorbed by the target nucleus and becomes excited because part of the kinetic energy was given to the nucleus as irritation energy. This scattering is called inelastic scattering and as in the fig(1).

2- Interaction of absorption: It occurs with slow neutrons where the neutron is absorbed by the nucleus and a compound nucleus is formed and the nucleus turns into an excited state called nuclear fission and as in the fig(2)[23].

3- Radiation capture of neutrons: The neutron is captured by the nucleus, becomes excited, and then emits gamma rays until it returns to a stable state. and symbolizes this interaction symbol (n,γ) and as in the fig(3)[24].

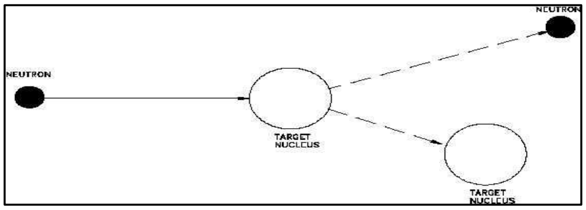


Fig.1: Elastic scattering.

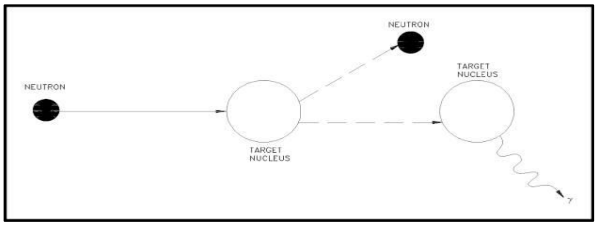


Fig.2: Inelastic scattering

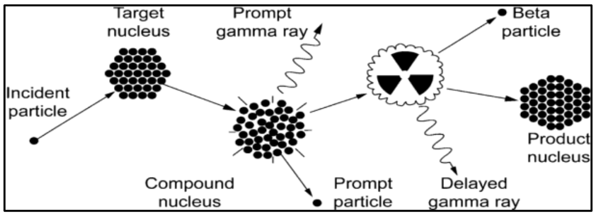


Fig.3: Neutron capture.

2-4 The Mechanism of Chemical Reaction

CdSe films were prepared using a chemical bath deposition CBD technique, which is based on the slow release of Se^{-2} and Cd^{+2} ions in solution and then condensed to deposition on a glass base placed inside the solution. The following equations represent the reaction mechanism to obtain CdSe films.

$$Se + Na_2SO_3 \longrightarrow Na_2SeSO_3$$

$$Na_2SeSO_3 + 2OH^- \longrightarrow Na_2SO_4 + H_2O + Se^{-2}$$

$$2CdCl_2 + 4NH_3 \longrightarrow (Cd(NH_3)_4)^{+2} + CdCl_4^{-2}$$

$$(Cd(NH_3)_4)^{+2} \longrightarrow Cd^{+2} + 4NH_3$$

$$Cd^{+2} + Se^{-2} \longrightarrow CdSe \downarrow$$

3 Results and Discussion

Optical, structures and morphological analysis of CdSe films were performed using UV-Vis spectrophotometer, XRD, and FESEM.

3.1 Optical Measurements

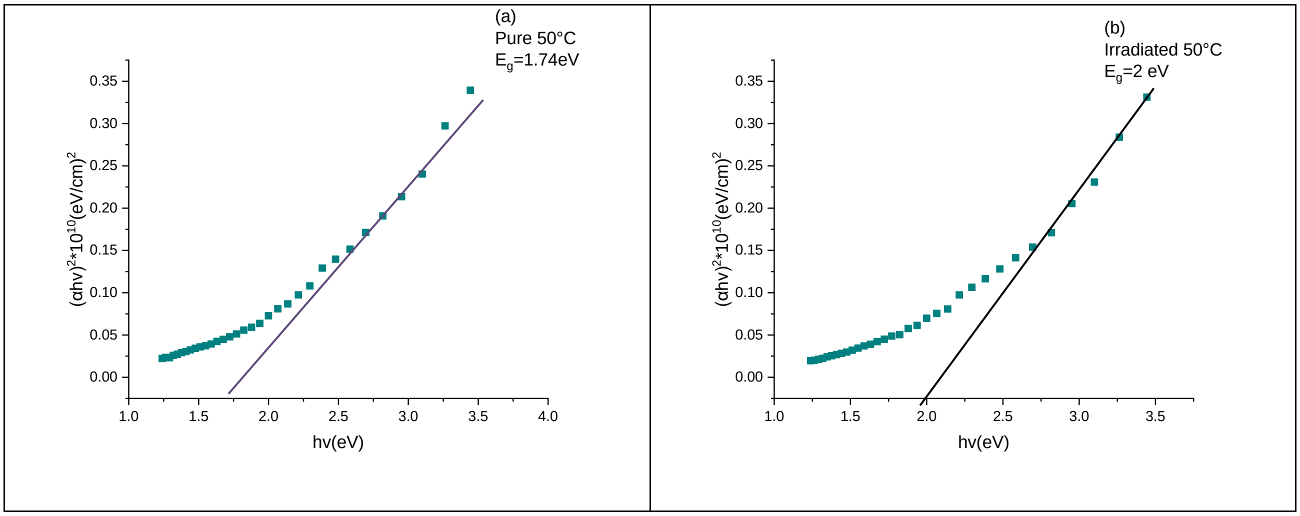
Figure 4 shows the optical absorption regions where the exponential region appears when the transitions are between the extended levels in the valence band to the local levels in the conduction band or from the local levels at the top of the valence band to the extended levels at the bottom of the conduction band [25].

Calculating the direct energy gap requires drawing the relationship $(\alpha h\nu)^2$ as a function of the photon energy(hν) where the extension of the straight part of the curve that falls on the axis of the photon energy represents the direct energy gap as shown in Figure (4).

Given the importance of the energy gap in the possibility of determining the use of thin films in the makings of solar cells and hybrid junctions because it is a basic measure for spectrum selection, Therefore, the values of the direct energy gap of this film were calculated before and after irradiation, and it was found from these values that the energy gap increased after irradiation.

The exposure of thin films to radiation doses may lead to an improvement in their behavior, as it was found that the values of optical properties increase with increasing radiation dose, and the energy gap value increases as a result of additional generation energy levels within the defined region between the equivalence and conduction band[26,27].

Figure 4, Shows the energy gap for pure CdSe films and irradiated Neutron.



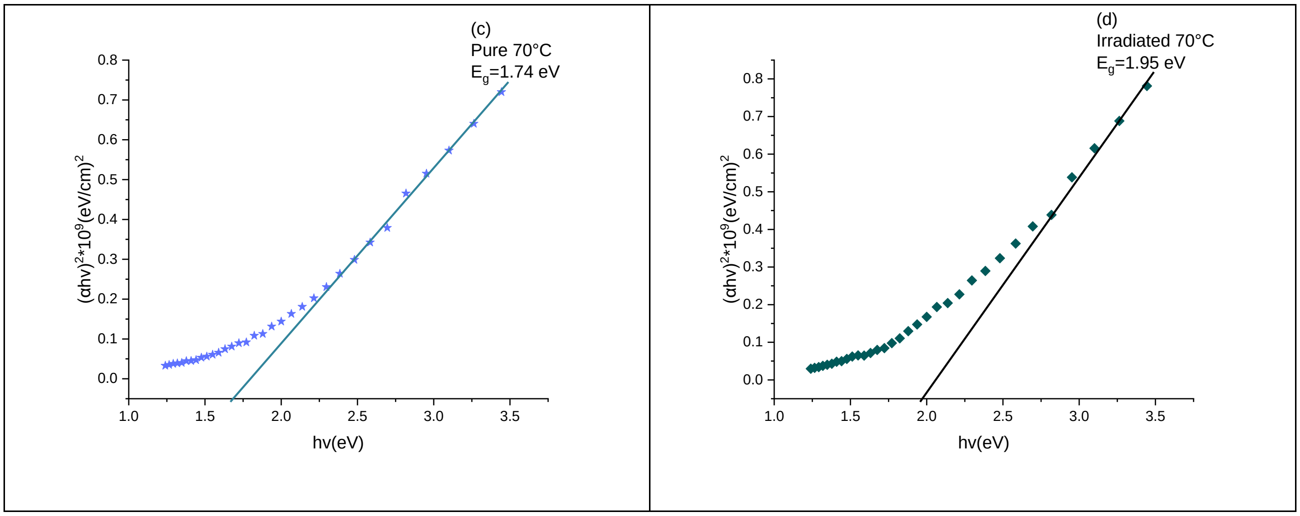


Fig. 4: Energy gap of CdSe thin films.

(a) pure at 50 °C (b) Irradiated at 50 °C (c) pure at 70 °C (d) Irradiated at 70 °C

Table 1: Thickness and energy gap for pure and neutron irradiated CdSe films

Temperature	**T**	**Before irradiation**	**After irradiation**	**Before irradiation**	**After irradiation**
C°	(μm)	**E_g(eV)**	**E_g(eV)**	**E_U(meV)**	**E_U(meV)**
50	0.3	1.74	2	740	800
70	0.4	1.74	1.95	833	909

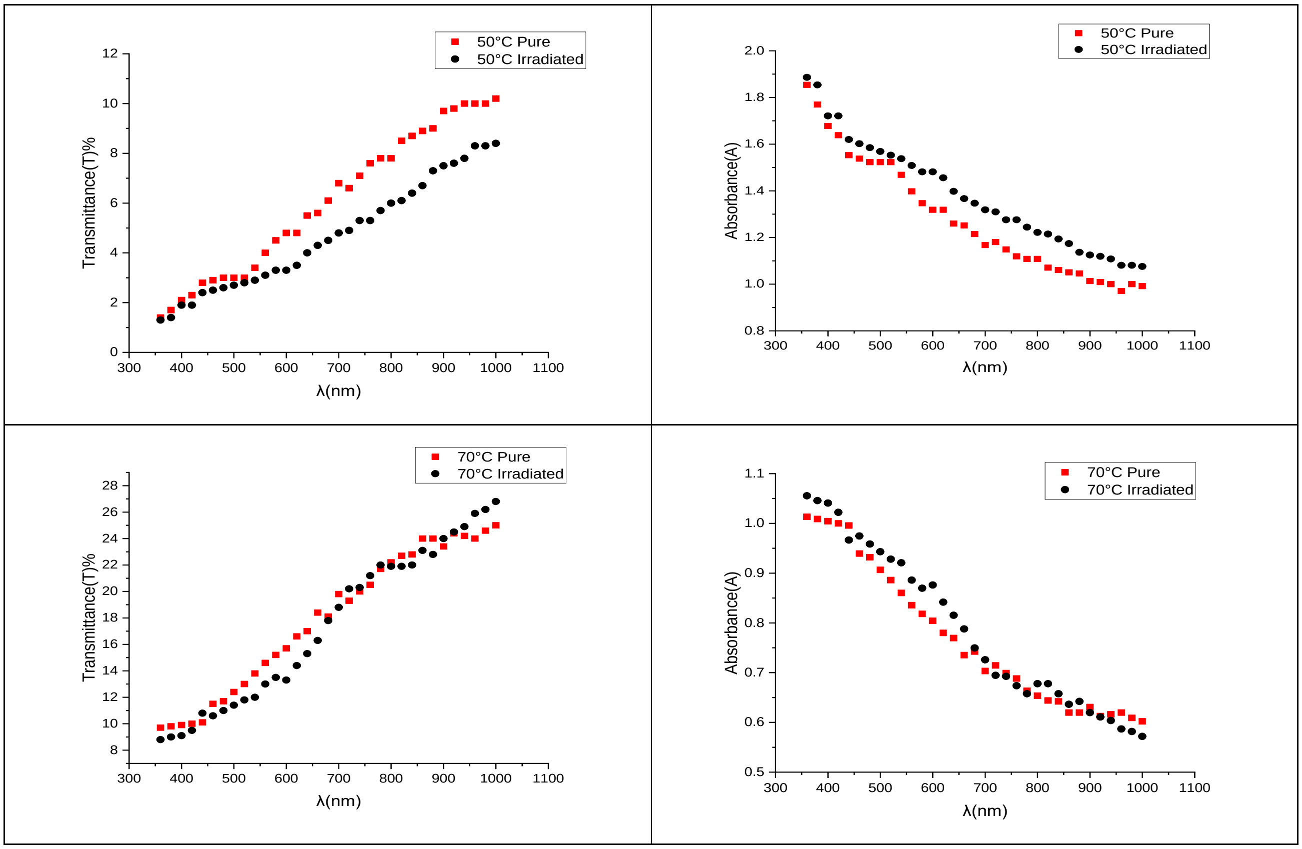


Fig. 5: Transmittance (T) and absorbance (A) as a function of wavelength (λ) for pure and irradiated CdSe films at 50°C and at 70°C

Table 1, shows the E_g and E_U values for CdSe thin film before and after exposed neutron irradiation at different temperatures.

It is found that the values before irradiation (1.74)eV and upon irradiation the energy gap increased and it is believed that the increase in the gap is due to the increase in the size of the grains. As shown in FESEM images Figure 10, it also increased the regularity of the internal arrangement of the atoms of the film. This is consistent with both[28,29] .

Figure 5, shows that CdSe films have low permeability in the ultraviolet region (340-400)nm and increase with increasing wavelength in the visible spectrum region (390 – 770)nm and the near-infrared (700 – 990)nm and this is consistent with what was stated by him and[7], and this indicates that CdSe films have a large energy gap that allows a part of the visible light to penetrate and upon irradiation, the permeability decreases and the absorbance increases due to an increase in the crystal size as well as an increase in the absorbance and optical absorption coefficient.

Urbach energy located in the exponential absorption region, and its value increases with the increase in the absorption of photons because the optical absorption of this region is associated with the transitions from the tails levels above the valence band to the extended levels in the conduction band or from the extended levels in the valence band to the states of tails below the conduction band.

The Urbach energy can be calculated by taking the reciprocal of the slope resulting from plotting the relationship between (Lnα) and (hν) [30]:

$$E_U = \left[\frac{d(ln\alpha)}{d(h\nu)}\right]^{-1}$$

When the samples are irradiated with a particular wavelength, these defect states trap the excited electrons, preventing their direct transition to the conduction band. These defect states are responsible for the absorption tail in the absorption spectra, which extends into the forbidden gap [31].

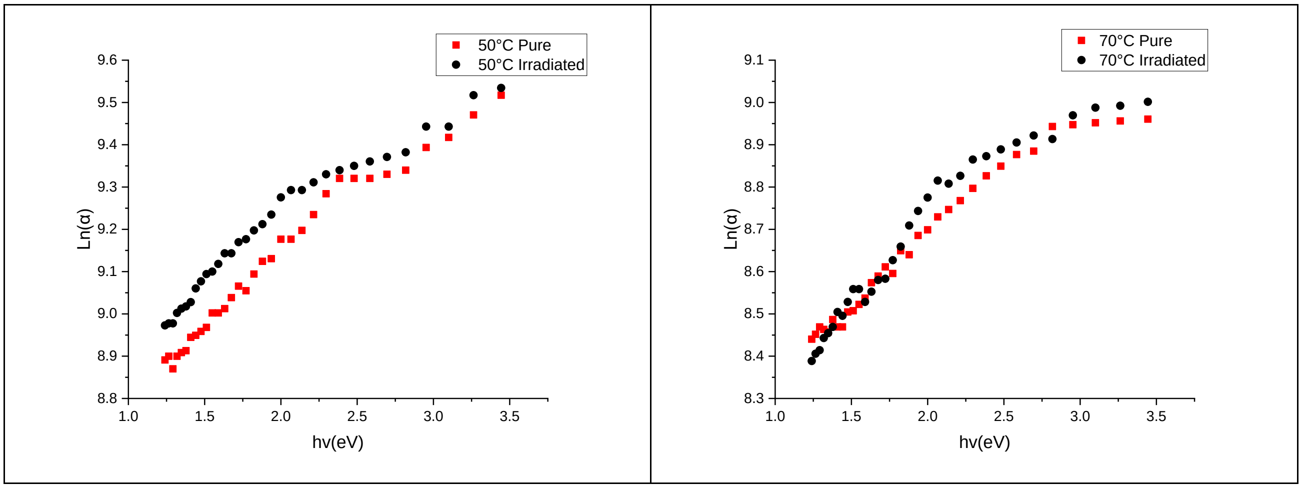


Fig. 6: Urbach energy for pure and irradiated CdSe films at 50^{o}C and 70^{o}C.

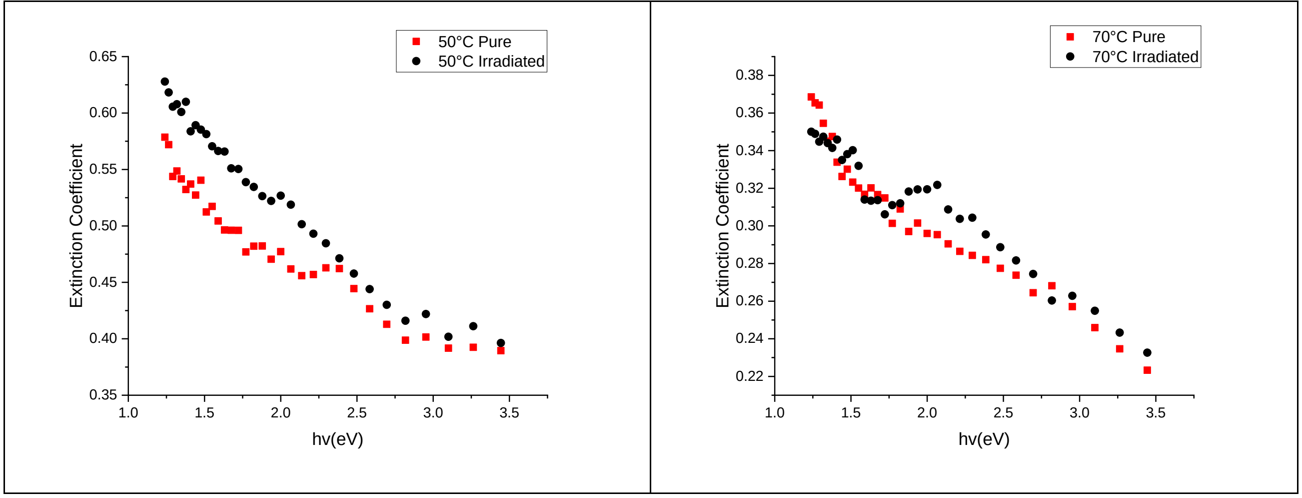


Fig. 7: The extinction coefficient(K) of CdSe films as a function of the photon energy before and after irradiation and at 50 and 70 °C.

Figure 6, shows An increase in E_U indicates the presence of a high density of levels centered inside the energy gap due to the formation of crystal defects within the film [32].

Figure 7, shows that the irradiation has affected the surface of the thin film, and this lead that the total dose absorbed by the film increases, and thus the defects left by the radiation on the film increase, which may lead to a defect in the crystalline structure of the film and this consistent with [33]. It was noted that the extinction coefficient increases with irradiation.

3.2 X-ray Diffraction Measurements (XRD)

Figure 8, shows the x-ray diffraction pattern of pure and irradiated CdSe. Here three main peaks were identified

of the line is at the middle of the highest peak value, θ is the angle between the incident and scattered X-ray.

(111) at 25.59,(220) at 42.50, and (311) at 50.14. All these peaks were matched with the standard data card of JCPDS no. (19-0191). Also, it was found that the pure and irradiated CdSe films were polycrystalline and have a cube structure. The effect of irradiation on films is observed through the relative intensity value of each peak, where the intensity decreases with irradiation, and the increase in the crystalline size of the CdSe films, and this may be attributed to the appearance of different crystalline forms, the structure of the films has been transformed slightly from the cube structure to the hexagonal structure at a temperature of 70°C, Such a phase transition may occur due to a change in the atomic configuration with the emergence of unknown peaks, we expect the formation of a new substance as shown in Table (2).

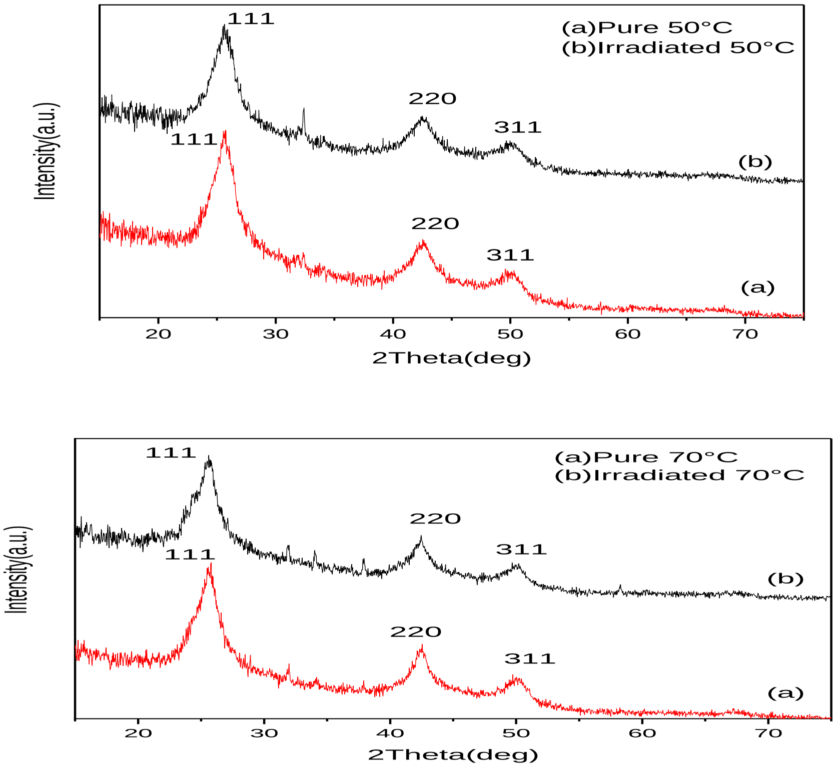


Fig. 8: XRD schemes of pure and irradiated CdSe films.

The crystallite size (D) of the CdSe film, was calculated from the Debye-Scherrer equation and it was found that the irradiation led to a narrowing of the peak width and thus a decrease in FWHM and thus the size increased.

$$D = \frac{K\lambda}{\beta COS\theta} ,$$

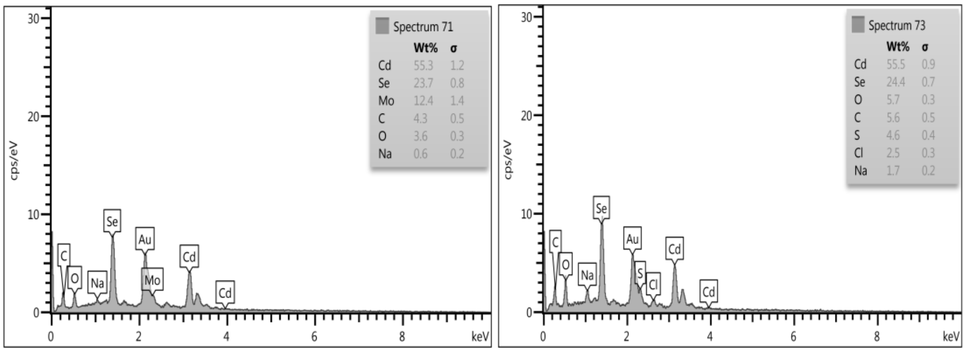
where k is a constant dependent on crystal type (0.9), λ is the wavelength of CuKα =1.54°A radiation, β is the width

3.3 Compositional Analysis

The elemental composition of CdSe films deposited on glass bases was performed using EDX analysis in Figure 9.

Table 2: Some of the factors obtained from the results of XRD of CdSe films pure and exposed to irradiation Neutron at a temperature of 50°C and 70°C.

Temp	**Model type**	**2θ**	**Peaks no.**	**Rel. Intensity**	**Hight**	**d-spacing**	**β**	**Crystallite Size**	**Ave. Size**	
C°		**(deg)**	**(hkl)**	**(counts)**		**A°**	**deg**	**(nm)**	**(nm)**	
50	Pure	25.59	(111)C	100	885	3.47797	2.10	4.09	4.09	CdSe
		42.50	(220)C	35.04	310	2.12528	2.5	4.03		CdSe
		50.14	(311)C	19.24	170	1.81805	2.7	4.15		CdSe
	Irradiated	25.62	(111)C	100	750	3.47418	2.26	3.81	4.78	CdSe
		31.94	(002)	15.27	115	2.80006	0.21	43.12		Cd
		32.364	ambiguous	38.40	288	2.76398	0.16	56.79		ambiguous
		42.45	(220)C	37.57	282	2.12780	2.36	4.27		CdSe
		49.98	(311)C	17.23	129	1.82344	2	5.60		CdSe
		67.7	(331)C	3.03	23	1.38214	3	5.45		CdSe
70	Pure	25.69	(111)C	100	779	3.46438	2.41	3.57	4.78	CdSe
		42.45	(220)C	44.57	347	2.12777	1.6	6.30		CdSe
		50.22	(311)C	24.66	192	1.81525	2.5	4.49		CdSe
	Irradiated	23.7	(100)H	15.30	103	3.74891	1	8.51	7.38	CdSe
		24.4	ambiguous	44.47	299	3.64313	1	8.57		ambiguous
		25.6	(111)C	100	673	3.47061	1	8.62		CdSe
		31.88	(002)	21.97	148	2.80496	0.14	64.48		Cd
		34.02	(100)	21.39	144	2.63307	0.12	76.66		Cd
		37.83	(101)	22.99	155	2.37635	0.02	242.10		Cd
		42.42	(220)C	41.93	282	2.12904	1.9	5.30		CdSe
		50.01	(311)C	22.62	152	1.82236	1.9	5.92		CdSe
		58.27	(104)H	11.48	77	1.58204	0.3	44.51		CdSe



a) **b)**

Fig. 9: The EDX pattern of CdSe films on the glass substrate at 50°C.

(a) Pure (b) irradiated

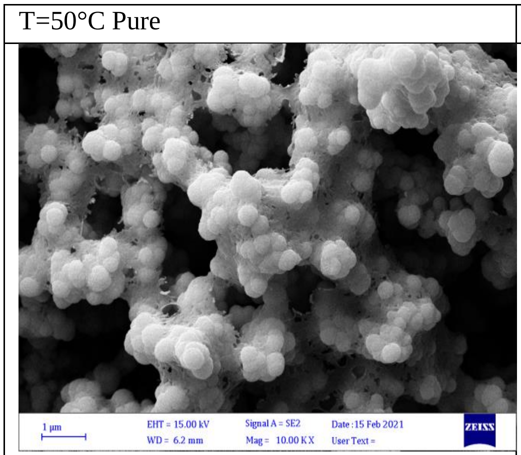
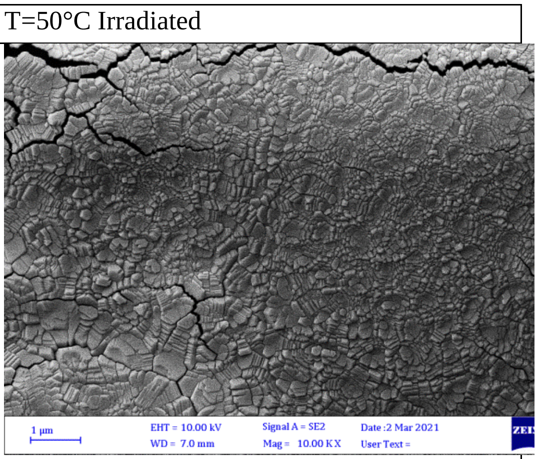
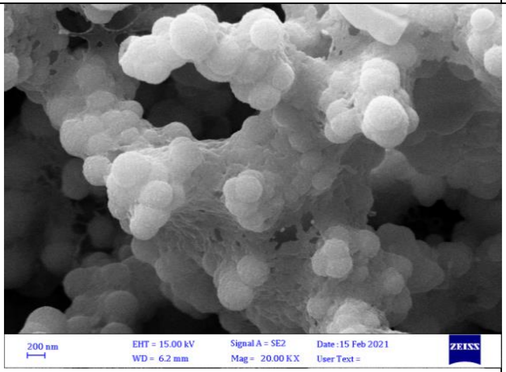
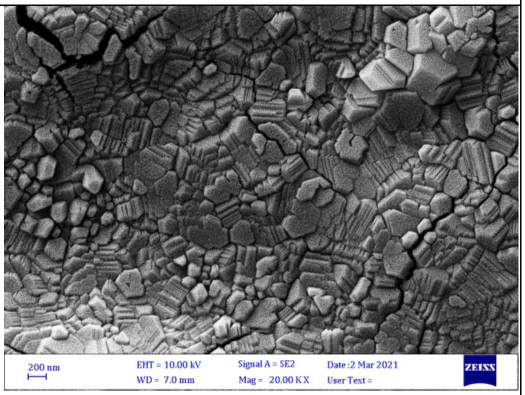
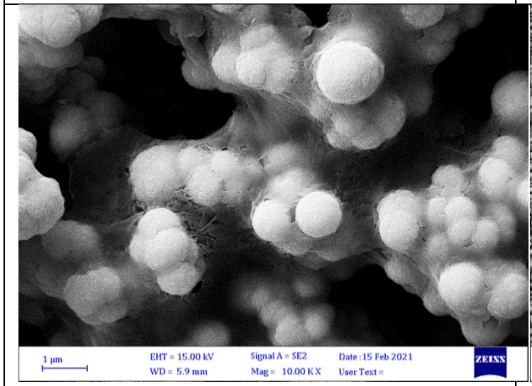
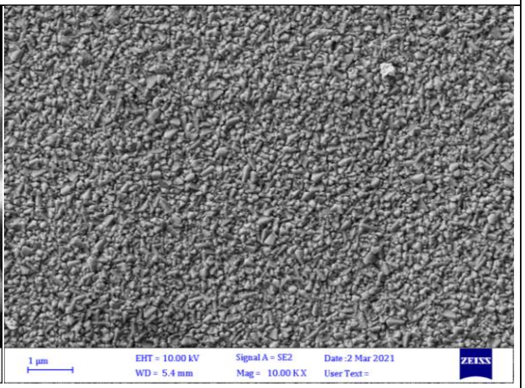
3.3 Compositional Analysis

The elemental composition of CdSe films deposited on glass bases was performed using EDX analysis in Figure 9. It is noted that there are some oxygen, carbon, and others, that can be attributed to the contamination of the film surface it might be from the experiment kit [34].

3.4 Morphological Surface

This technique is useful to determine the morphology of the surface. The obtained results show that CdSe films consist of uniform and homogeneous spherical granules without merging with some distances between them without cracks or holes, and after the irradiation, Cracks occurred at a temperature of 50°C and the spherical shape of the granule disappeared completely at temperatures 50°C and 70°C.

When the neutrons interact with the material, the large energy of the neutrons is given to the crystals, causing a rise in their temperature, forming a local fluid for the crystals, which transforms the crystals into random crystals and causes damage to the crystal lattice [35].

T=50°C Pure	T=50°C Irradiated
	
	
T=70°C Pure	T=70°C Irradiated
	

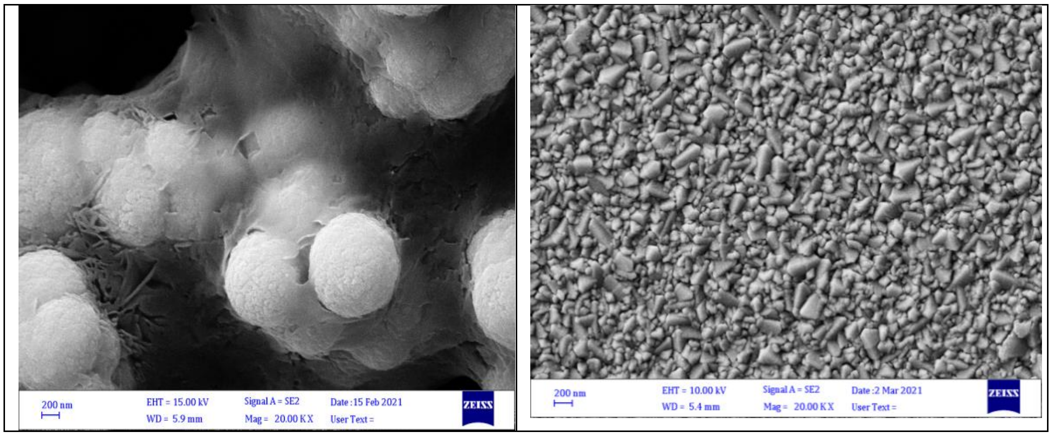


Fig. 10: FESEM image of as-synthesized CdSe nanoparticles by CBD.

4 Conclusions

Through this research, CdSe films were obtained at a temperature of 50°C and 70°C, PH = 9 and for a deposition period of 3 h, then irradiation by neutron beam from the source (Am^{241} – Be^{10}).

When studying the effect of irradiation on its structural, optical, and surface properties, it was found from the UV - V measurement that the energy gap increases with irradiation, the absorbance increases, the extinction coefficient increases with irradiation, and this proves that exposure of the film for a long period to radiation may cause a change in the nature of the surface of the film, which leads to damage to the crystal lattice. Urbach energy increases by irradiation and the transmittance decreases, the analysis of XRD showed that the pure CdSe films were polycrystalline with cubic structure, while unknown peaks appeared upon irradiation and at a temperature of 70°C the structure of the films was transformed from cubic to hexagonal structure and the particle size increases, while the FESEM measurements showed that there is a change in the shape of the sample surface if the shape of the spherical grains changes into compact, polygonal and coherent blocks as if they were subjected to momentum due to the neutron flux, and the film surface suffered sporadic cracks at 50°C, this increases the surface area of the CdSe film and its agglutination.

Acknowledgement

The researcher extends his sincere thanks to Dr. Naz Talab and researcher Mohammad Abdulqader Ibrahim for their assistance for neutron irradiation at the College of Education for Pure Sciences / Ibn Al-Haitham.

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