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OPTICAL PROPERTIES OF TIN OXIDE THIN FILMS BEFORE AND AFTER THERMAL AND FAST NEUTRON IRRADIATION

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ABSTRACT

This study investigates the effect of thermal and fast neutrons on the optical properties of a tin oxide (SnO_2) thin film, which was prepared by thermal evaporation in a vacuum. The thickness of the film ranged from 90 to 110 nm. The samples were irradiated using an americium–beryllium (Am–Be) neutron source, providing thermal neutrons (≈0.025 eV) and fast neutrons (≈5 MeV), with a flux of $3x10^5$ n/cm²s. The optical properties of the thin film were examined before and after the irradiation. Before irradiation, the energy gap (E_6) of the film was 3.307 eV. After one week of irradiation with thermal neutrons, the energy gap decreased to 3.259 eV, and after two weeks, it further decreased to 3.217 eV. For fast neutrons, however, the results indicated that the energy gap remained unchanged even after 14 weeks of irradiation. Regarding the transmittance, it was observed to increase with longer irradiation periods by thermal neutrons in the wavelength range from 190 to 686 nm. In comparison, no change was observed for wavelengths longer than 686 nm. For fast neutrons, no significant changes were observed in transmittance, likely because the cross-section of interaction between tin and fast neutrons is much smaller compared to the interaction between tin and thermal neutrons. Absorbance showed an inverse trend relative to transmittance and remained unaffected by fast-neutron irradiation. The behaviour of absorbance was similar to that of reflectivity before and after irradiation. As for the extinction coefficient (K_o), it was found to increase with thermal neutron irradiation, while fast neutrons did not affect it.

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1. INTRODUCTION

The term thin film is used to describe a layer or multiple layers of material whose thickness does not exceed one micrometre [1,2]. Thin films are prepared using various techniques that depend on the type of membrane and its intended use [3]. The most prominent types of these thin films are the transparent conductive oxide films, symbolized by the abbreviation TCO, such as tin dioxide (SnO2), zinc oxide (ZnO), and indium trioxide (In2O3) films. The most important characteristic of TCO thin film is their high transmittance to visible light and their good electrical conductivity, and this is what qualifies them for their independence in many applications, which have contributed to the development of digital computers, copy devices, and photography. as well as window coatings, their high reflectivity for infrared rays, and also in the manufacture of thermal, gas, optical sensors and other applications [4 – 6]. Tin dioxide has a high melting point of up to 1063°C, which makes it difficult to prepare. Tin dioxide is also considered a negative-type semiconductor. Tin dioxide is prepared by heating the tin in the presence of aqueous oxide, which results from the reaction of metallic tin with concentrated nitric acid [7,8]. It was found that the tin dioxide film prepared by thermal chemical spraying at different temperatures is amorphous at temperatures below 220°C and crystalline in the range of 300 to 500°C [9]. In this work, a tin dioxide thin film was irradiated with thermal and fast neutrons at different doses to determine the extent of this film's response to the irradiation process by studying the optical properties of the film before and after irradiation, such as transmittance, absorbance, reflection coefficient, extinction coefficient, and optical energy gap. This work supports previous studies in references [10,11] on tin oxide, which focused on gamma irradiation. The work further explores the effects of radiation, particularly thermal and fast neutrons, on the optical properties of tin oxide films. Most of the published material, as detailed in the references, is limited to gamma irradiation [12–14]. Practical studies on neutron irradiation are limited, as most research in this area consists of simulations. Recent works have highlighted the importance of neutron–matter interactions and their impact on defect formation and optical behavior in oxide materials [15-17]. However, most of these studies are either limited in scope or based on simulations, indicating a clear need for systematic experimental investigations. Therefore, there is a clear need for systematic experimental studies to understand the influence of neutron irradiation on the optical properties of SnO_2 thin films. In this work, SnO_2 thin films were irradiated with both thermal and fast neutrons at different doses, and their optical properties—including transmittance, absorbance, reflectance, extinction coefficient, and optical energy band gap—were investigated before and after irradiation. This study aims to bridge the gap in the literature by providing experimental insight into neutron-induced modifications in thin film materials.

The paper is organised as follows: Section 1 presents a literature review and general information about the paper. Section 2 describes the experimental part used in the calculation, and Section 3 presents the study's findings. Finally, concluding remarks are presented in Section 4.

2. MATERIALS AND METHODS

2.1. Sample Preparation

The samples were prepared at Tikrit University Nuclear Physics Laboratory using a vacuum thermal evaporation system, as shown in Figure 1. Tin oxide powder was first ground using an agate mortar and then placed in an iron mold. The powder was compacted using a hydraulic press under a pressure of 5 tons for one minute, with a mold area of 5 mm. The compacted material was then plastified at a temperature of 100 °C and subsequently deposited using a vacuum thermal evaporation apparatus. A molybdenum (Mo) crucible was used during the evaporation process to avoid oxidation. After deposition, the films were annealed in a furnace at 450 °C for two hours. The thickness of the deposited tin oxide thin films ranged from 90 to 110 nm. The prepared films exhibited a tetragonal crystalline structure, with tin oxide particles showing a sand-like morphology and particle sizes in the range of 10–30 nm

2.2. Irradiation Process

The deposited thin films were irradiated in contact with a 241Am – 9Be neutron source with a flux of ($3.5\times10\text{^}5$ $n.s^{-1}.cm^{-2}$), emitting fast neutrons of approximately 5 MeV. The unit ($n.s^{-1}.cm^{-2}$) refers to flux, not activity. The irradiation system is available at the College of Education for Pure Sciences, Ibn Al-Haitham, University of Baghdad. Thermal neutrons with an energy of approximately 0.025 eV were produced by surrounding the source with a paraffin wax moderator, as shown in Figure 2. For radiation protection, additional shielding materials such as sand and water were used alongside the paraffin wax. The tin oxide thin films were irradiated for durations of one and two weeks.

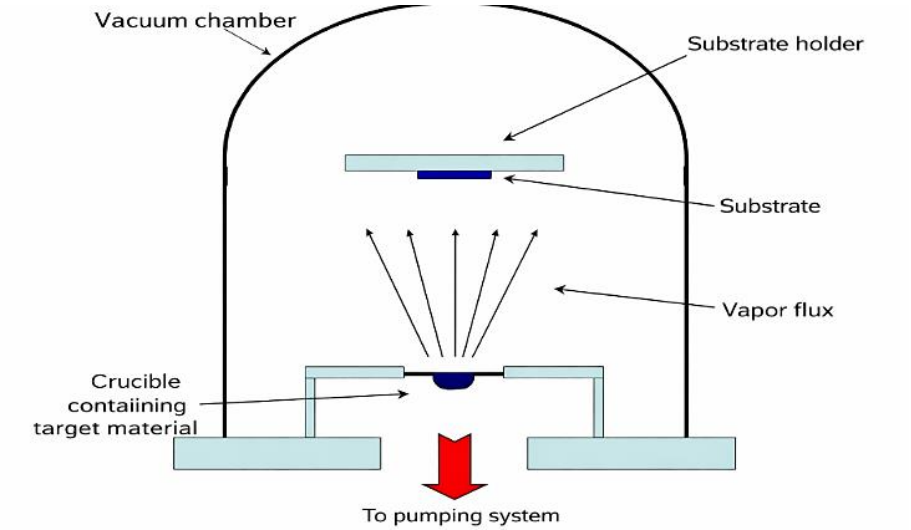
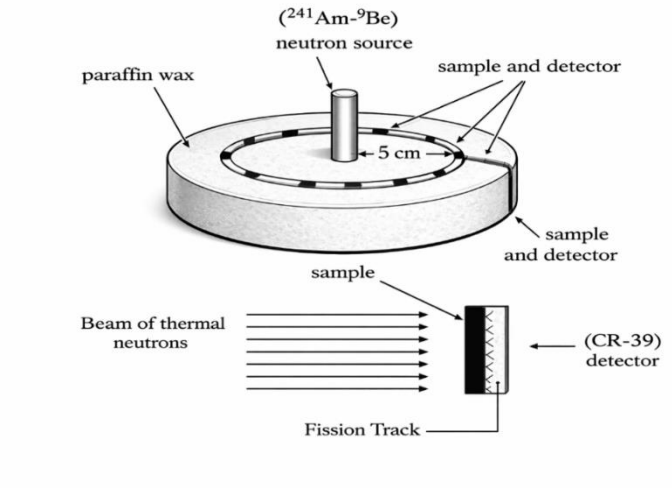
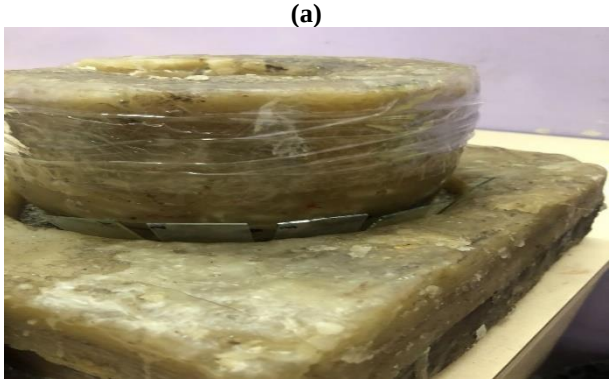


Figure 1. *Thermal evaporation chamber*



(a)



(b)

Figure 2. (a) *Schematic diagram illustrating the irradiation configuration of SnO_2 thin film samples.* ***(b)*** *Actual experimental setup used for irradiation measurements*

Figure 2(a) illustrates the schematic representation of the neutron irradiation and detection setup, while Figure 2(b) shows the actual experimental system. A ^{241}Am–^{9}Be neutron source positioned at the center emits fast neutrons, which are subsequently moderated to thermal energies by the surrounding paraffin wax. The moderated neutrons interact with the sample placed within the irradiation field. Induced fission reactions generate charged particles that leave latent damage tracks in the CR-39 solid-state nuclear track detector. These tracks can later be chemically etched and analyzed to determine neutron-induced fission events and material characteristics.

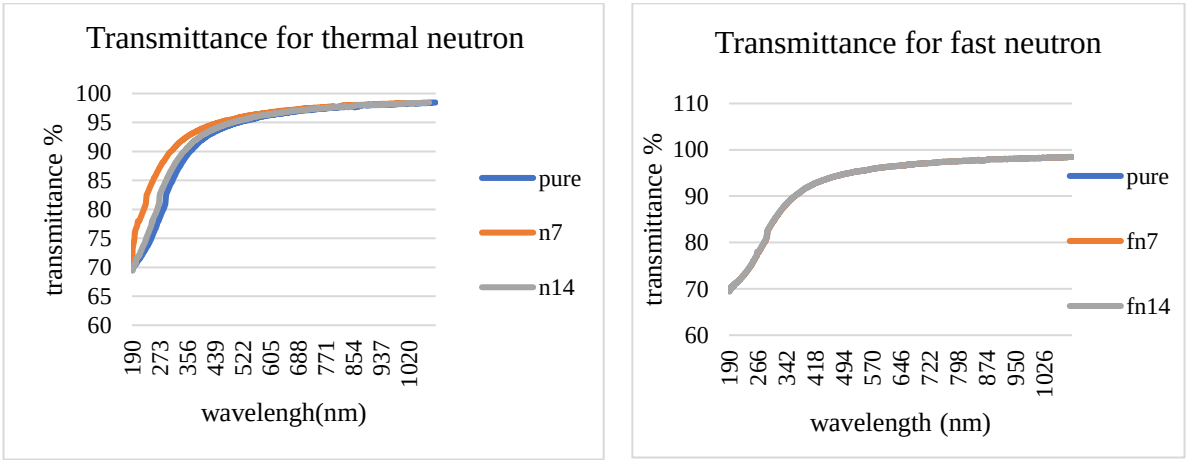
2.3. Measurements

A series of measurements was conducted on both non-irradiated and irradiated thin films. All optical measurements were performed at the College of Science, Tikrit University, Iraq, including the determination of the optical energy band gap for allowed direct transitions and transmittance measurements in the wavelength range of 190–1100 nm using a UV–Vis spectrophotometer. The experimental procedure included the determination of the optical energy band gap for allowed direct electronic transitions, transmittance and absorbance measurements in the wavelength range of 190–1100 nm, calculation of the absorption coefficient, reflectance measurements, and structural characterization using X-ray diffraction (XRD) analysis. In addition, the variation of transmittance and absorbance as a function of wavelength in the range of 300–1100 nm was analyzed to evaluate the optical properties of all samples.

3. RESULTS AND DISCUSSION

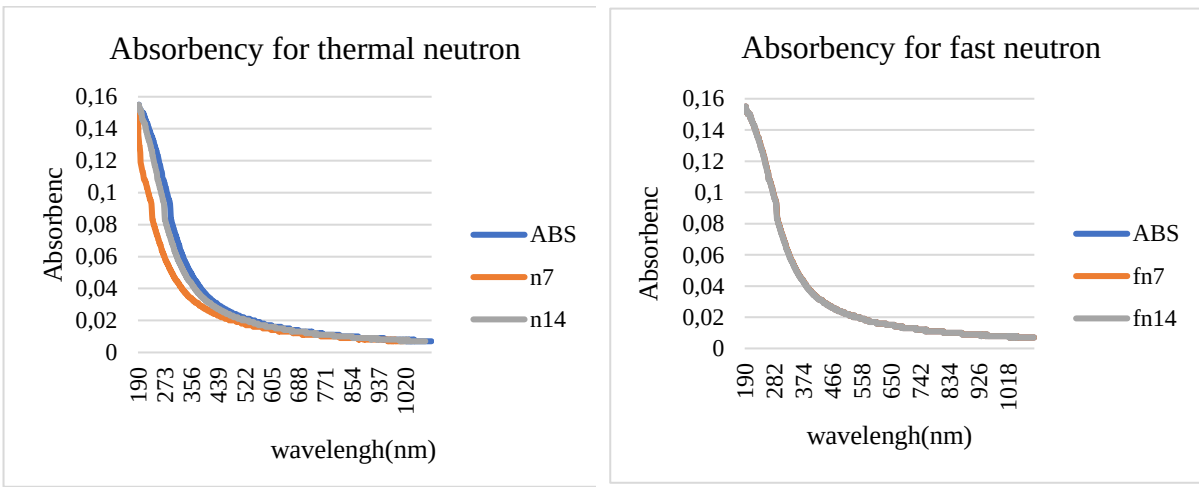
Firstly, the optical properties of the SnO_2 thin films were systematically investigated before and after thermal and fast neutron irradiation to evaluate the effects of neutron exposure on their optical behavior. The optical characterization included transmittance, absorbance, optical band gap, absorption coefficient, reflectance, and X-ray diffraction (XRD) analyses. The obtained results are comparatively discussed to reveal the structural and optical modifications induced by neutron irradiation. Figure 3(a) presents the variation of transmittance as a function of wavelength in the range of 190–1100 nm for the pristine SnO_2 thin film and the samples irradiated with thermal neutrons (0.025 eV). As can be seen from Figure 3(a), the transmittance spectra exhibit noticeable changes after neutron irradiation, indicating that thermal neutron exposure influences the optical response of the SnO_2 thin films. The thin film was irradiated with a flux of $2.117x10^{11}$ and $4.23x10^{11}$ $n.s^{-1}.cm^{-2}$, so the transmittance was higher than 95% in the spectral range from 539 to 1100 nm, where there is no difference between the pure sample and the one irradiated with fast neutrons, as shown in Figure 3 (b). Figure 3 shows that the transmittance increases with irradiation with thermal neutrons, with the first dose doing more than the second dose. It appears that the transmittance of the non-irradiated sample did not change for all ranges of the spectrum, as is the case for thermal neutrons. This means that there is no interaction with the fast neutrons because the cross-section adsorption with them is small ($\sigma a = 0.001$ barn), like thermal neutrons, which is ($\sigma a = 0.626$ barn). This result is in agreement with reference [11] about increasing the transmittance with increasing irradiation by gamma and neutrons. Secondly, the absorbance and reflectivity were plotted as a function of wavelength, as shown in Figures 4 (a), 4 (b), 5 (a) and 5 (b). This showed the same behaviour for both of them, which is opposite to the behaviour of the transmittance function for wavelengths in the ranges from 190 to 686 nm, and for more than 686 nm. There were stable values for the three cases. Third, the reflection coefficient was also plotted with wavelengths in Figures 6 (a) and 6 (b), which shows the fluctuation in the reflection coefficient values at short wavelengths, in addition to the decrease in the absorption coefficient upon irradiation. The highest value was at the wavelength of 316 nm for the pure sample and the sample irradiated with thermal neutrons, the second dose. As for the sample irradiated with the first dose, the highest value of the reflection coefficient was at a wavelength of 232 nm, where it fluctuated for some values, while the reflectivity coefficient was not affected when irradiated with fast neutrons. The maximum value of the reflection coefficient was approximately at a wavelength of 316, and it fluctuated before this range, but beyond this range, it declined as an exponential function. Figure 7 (a) shows the relationship of the extinction coefficient as a function of wavelength, where we find the highest value for the pure model at 286 nm and for the model irradiated with the second D2 thermal neutrons at 332 nm. As for the first dose of neutrons, we find it at 256 nm and before this range, there is a dimension of oscillation at wavelengths less than 330 nm. As for Figure 7 (b) for the fast neutron model, the results showed that the highest value of the extinction coefficient was at 292 nm for the pure model and at 351 nm for the radioactive model with the first dose and the second dose. Figures 8–12 illustrate the optical energy band gap of SnO_2 thin films in both non-irradiated (pure) and neutron-irradiated conditions, obtained from Tauc plots of $(\alpha h\nu)^2$ versus photon energy $h\nu$, assuming allowed direct transitions. In these plots, the photon energy $h\nu$(eV) is represented on the x-axis, while $(\alpha h\nu)^2$(a.u.) is plotted on the y-axis. The optical band gap values were determined by extrapolating the linear region of each curve to the energy axis. Figure 8 shows that the optical band gap of the pure SnO_2 film is approximately 3.307 eV. After irradiation with thermal neutrons for 7 days (Figure 9), the band gap slightly decreases to about 3.259 eV, and a further reduction to approximately 3.217 eV is observed after 14 days of irradiation (Figure 10). This decrease can be attributed to the formation of defect states and increased structural disorder induced by neutron irradiation, which introduces localised energy levels within the band gap. For fast neutron irradiation, a slightly different behavior is observed. As shown in Figure 11, the band gap decreases to about 3.233 eV after 7 days of irradiation. However, after 14 days (Figure 12), the band gap increases to approximately 3.366 eV. This increase may be associated with defect reorganization, partial recovery of crystallinity, or changes in carrier concentration, which can lead to band gap widening, possibly due to the Burstein–Moss effect. Overall, the optical energy gap values before and after neutron irradiation remain relatively close, showing slight oscillatory behavior depending on irradiation type and duration.

However, in most cases, the band gap values after irradiation are lower than those of the non-irradiated sample. This behavior may be attributed to irradiation-induced modifications in the material structure, including defect formation and possible phase changes, as clearly observed in Figures 8–12. The chemical composition of the studied film is tin dioxide (SnO_2). For thermal neutrons, the absorption cross-section of tin (Sn) is approximately 0.6–1 barn, while the scattering cross-section is about 4–6 barn. In contrast, oxygen (O) exhibits a negligible absorption cross-section (~0.0002 barn) and a scattering cross-section of about 3–4 barn. Accordingly, the total cross-section of SnO_2 is estimated to be in the range of 5–8 barn. Since absorption is relatively weak compared to scattering, the material can be considered semi-transparent to thermal neutrons. For fast neutrons (with energies > 1 MeV), the absorption cross-section of tin decreases to approximately 0.1–0.3 barn, while the scattering cross-section is about 2–4 barn. Oxygen again shows negligible absorption and a scattering cross-section of approximately 2–3 barn, resulting in a total cross-section in the range of 2–5 barn. Therefore, absorption is further reduced and elastic scattering becomes dominant. This behavior is consistent with the X-ray diffraction (XRD) results of the samples, as shown in Figures 13 and 15, where irradiation-induced structural modifications are observed. The X-ray diffraction (XRD) patterns of the SnO_2 thin film samples are presented in Figures 13 – 15. The diffraction intensity (a.u.) is plotted as a function of the diffraction angle 2θ (degrees). The broad features observed in the patterns indicate the structural characteristics of the films, while variations in intensity suggest changes in crystallinity and possible effects of irradiation. The indicated regions highlight noticeable changes in the diffraction profiles, which may be associated with structural modifications in the thin films. This showed great randomness as a result of neutron irradiation, which leads to great scattering of light. Where the structure is regular before irradiation.



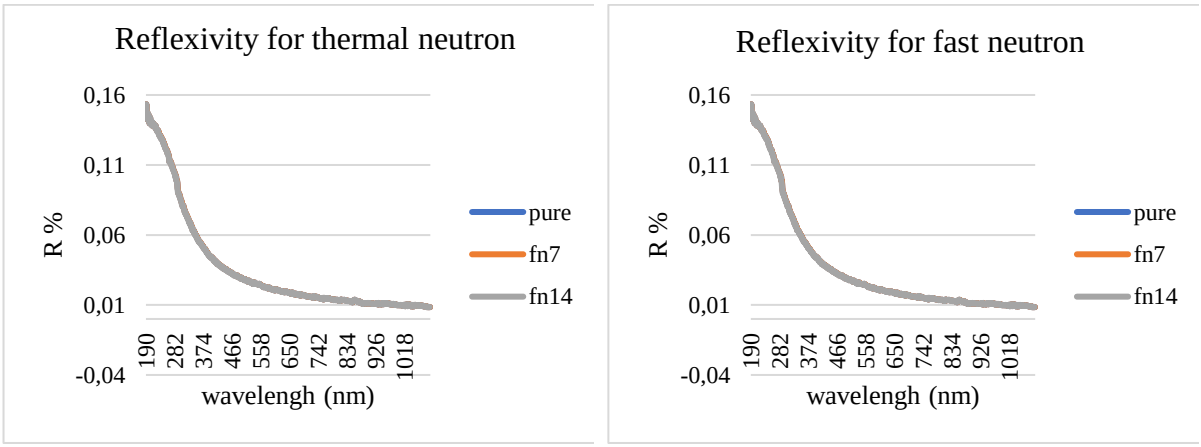
(a) **(b)**

Figure 3. *The transmittance of SnO_2 film irradiated with* ***(a)*** *thermal neutron,* ***(b)*** *fast neutron.*



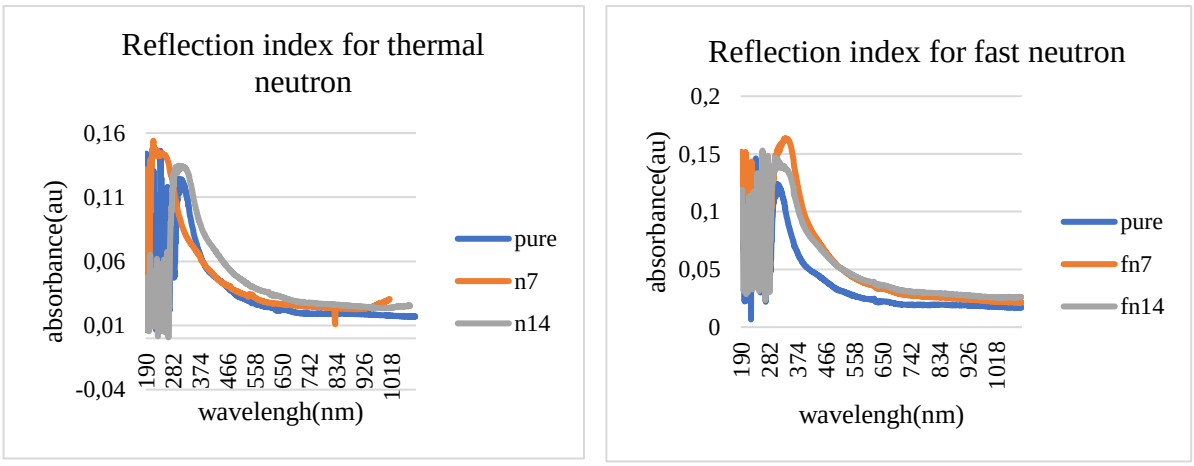
(a) **(b)**

Figure 4. *The absorbency of SnO_2 film irradiated with* ***(a)*** *thermal neutron,* ***(b)*** *fast neutron.*



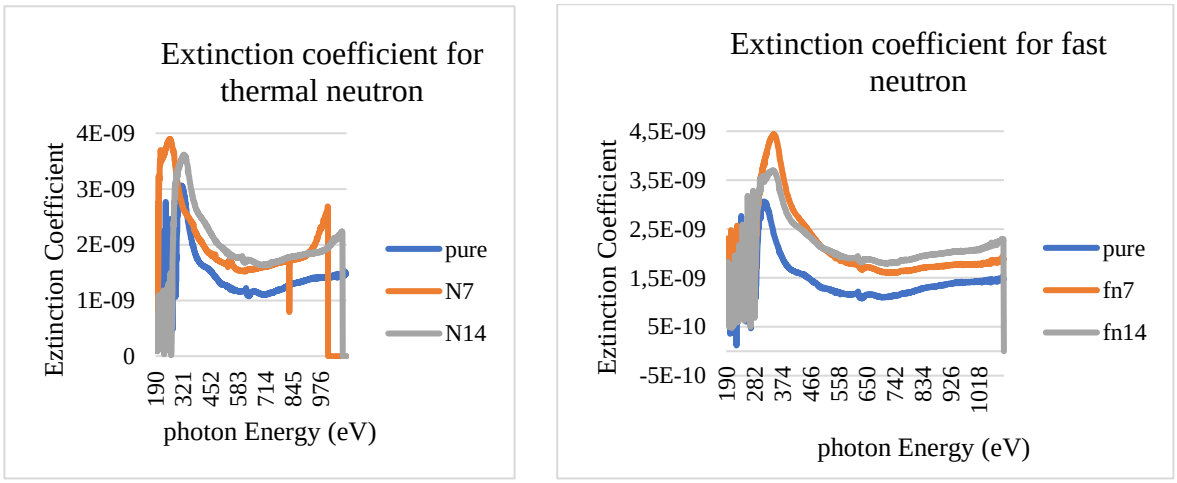
(a) **(b)**

Figure 5. *The reflexivity of SnO_2 film irradiated with* ***(a)*** *thermal neutron,* ***(b)*** *fast neutron.*



(a) **(b)**

Figure 6. *The reflection index of SnO_2 film irradiated with* ***(a)*** *thermal neutron,* ***(b)*** *fast neutron.*



(a) **(b)**

Figure 7. *The extinction coefficient of SnO_2 film irradiated with* ***(a)*** *thermal neutron,* ***(b)*** *fast neutron.*

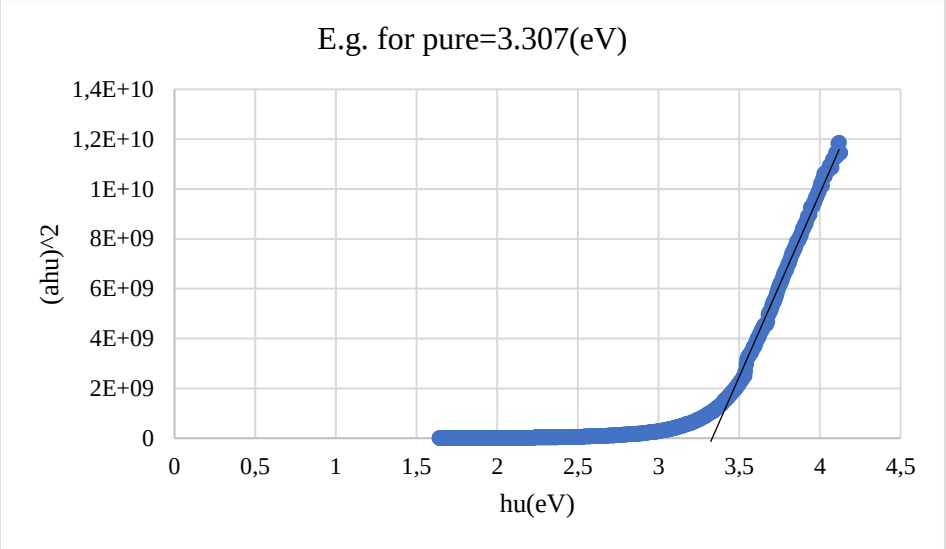


Figure 8. *The optical energy gap of SnO_2 film without irradiation (pure).*

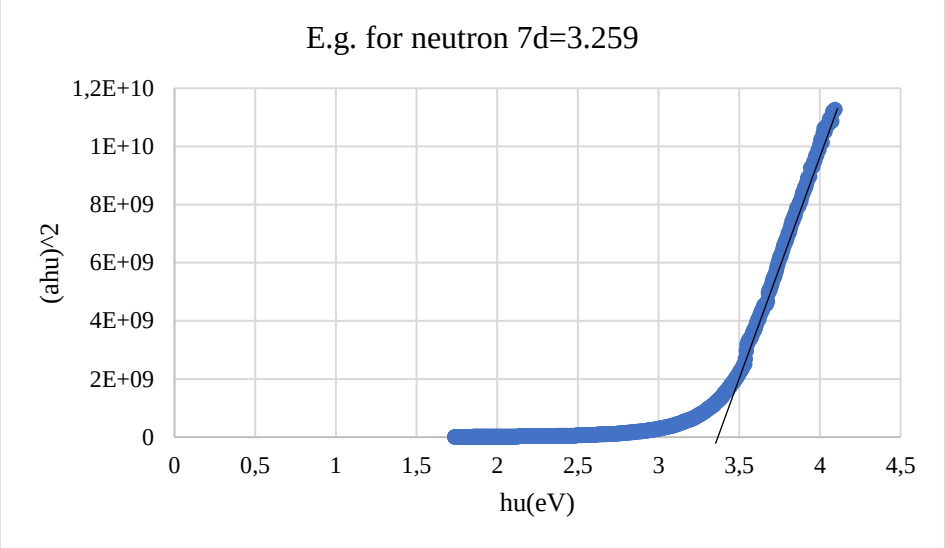


Figure 9. *The optical energy gap of SnO_2 film irradiated with thermal neutrons (7 days).*

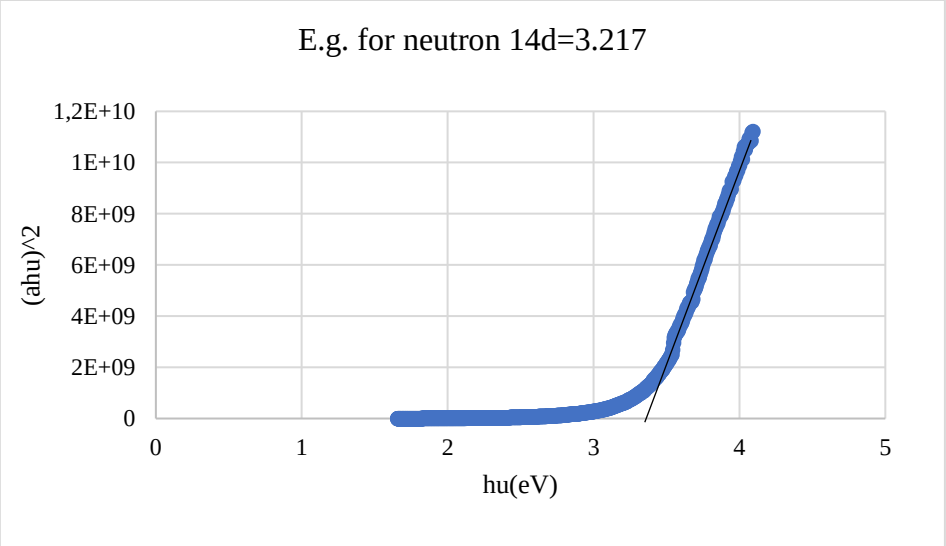


Figure 10. *The optical energy gap of SnO_2 film irradiated with thermal neutrons (14 days).*

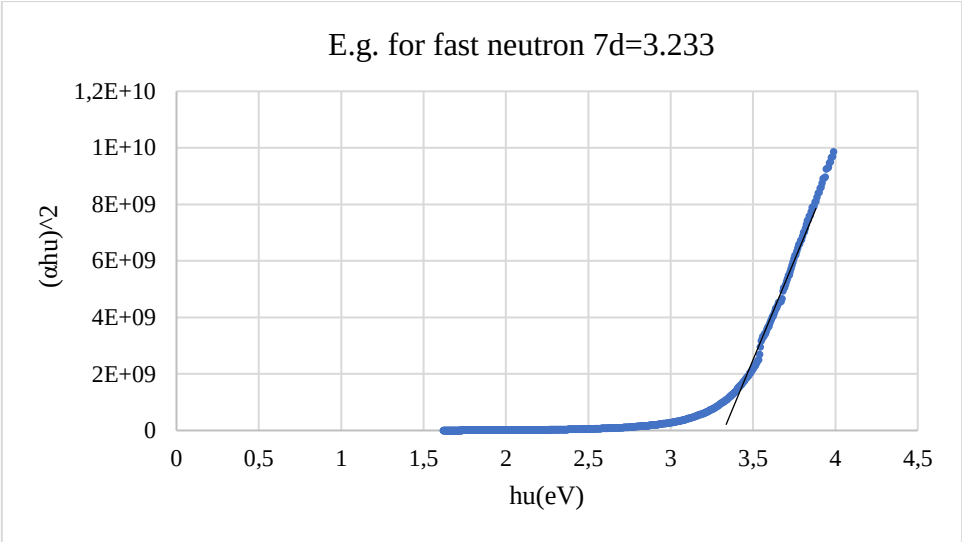


Figure 11. *The optical energy gap of SnO_2 film irradiated with fast neutrons (7 days).*

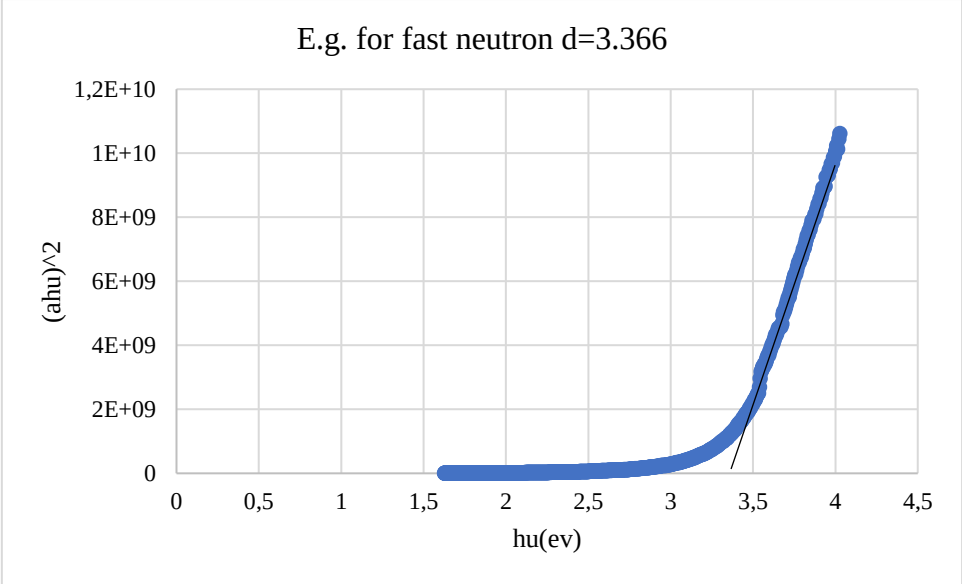


Figure 12. *The optical energy gap of SnO_2 film irradiated with fast neutrons (14 days).*

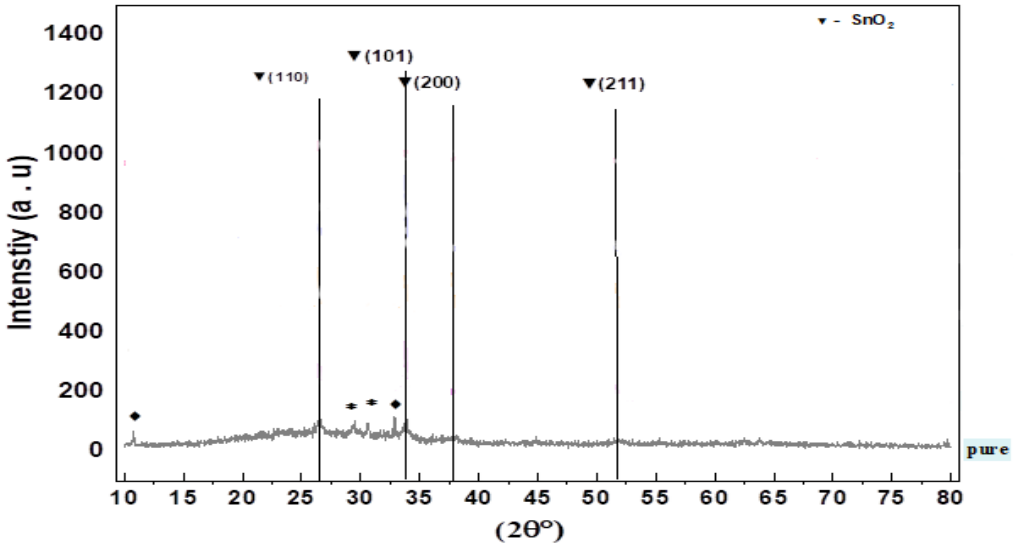
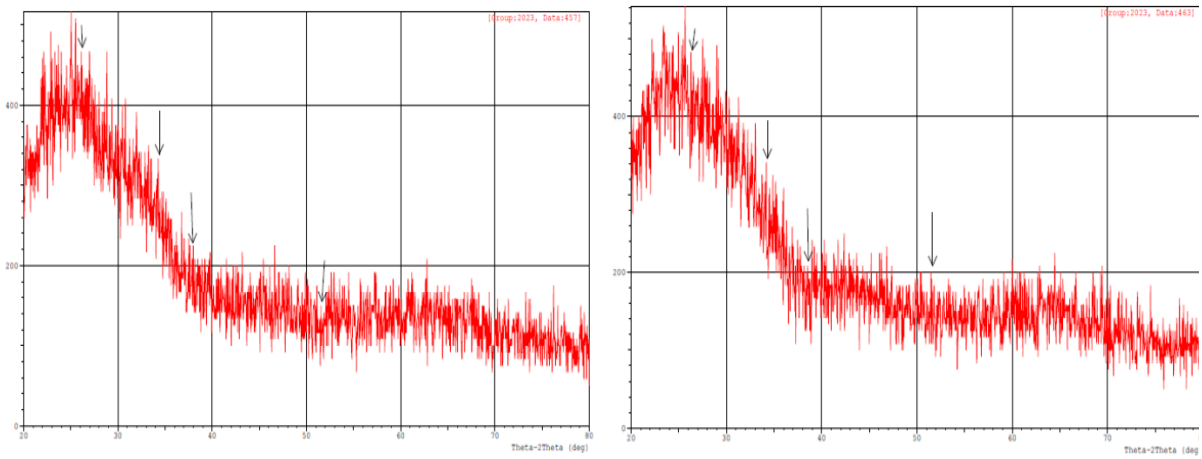
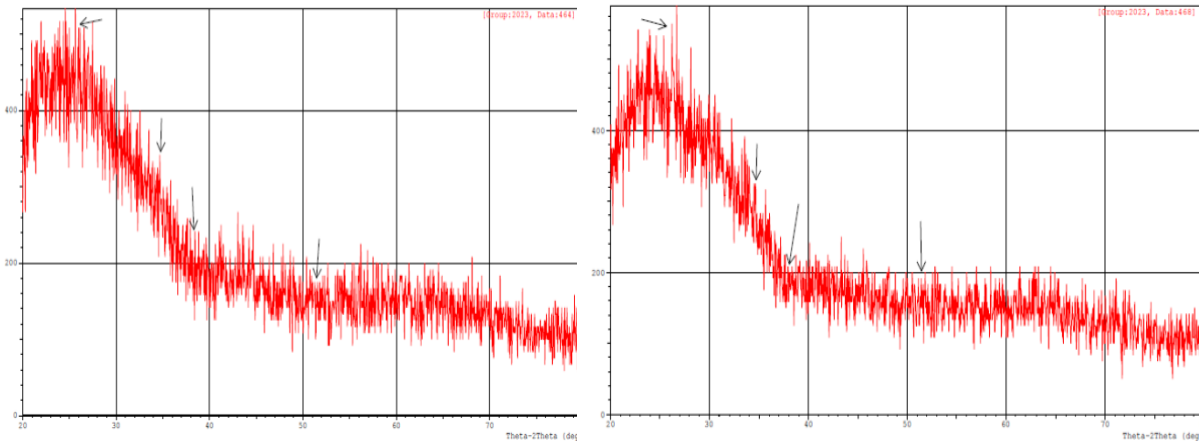


Figure 13. *The X-ray diffraction of SnO_2 film without irradiation (pure)*



(a) **(b)**

Figure 14. *The X-ray diffraction of SnO_2 film irradiated with thermal neutrons,* ***(a)*** *7 days* ***(b)****14 days.*



(a) **(b)**

Figure 15. *The X-ray diffraction of SnO_2 film irradiated with fast neutrons,* ***(a)*** *7 days* ***(b)****14 days.*

4. CONCLUSIONS

In this work, the effects of neutron irradiation on SnO_2 thin films were systematically investigated. The results show that thermal neutron irradiation, considering the significant absorption cross-section of tin (~6.8 barns), induces noticeable structural and optical modifications. The films exhibit a transformation from a polycrystalline to amorphous structure at low irradiation doses (7 days), followed by a transition toward a monocrystalline structure with increased dose and irradiation time (14 days). These structural changes are reflected in the optical properties, including transmittance, absorbance, reflectance, absorption coefficient, and extinction coefficient. Furthermore, a reduction in the optical band gap was observed after irradiation, which may be attributed to irradiation-induced phase changes. Overall, the pronounced response of the films to neutron irradiation suggests their potential applicability in radiation detection, particularly as thermal neutron sensors, in agreement with previous studies [10–12].

Conflict of Interest

There is no conflict of interest between the authors.

Authors Contributions

Necla Çakmak and Ulvi Kanbur conceived and designed the study, coordinated the research, drafted the manuscript, and performed the statistical analysis. Khalid Hadi Mahdi Aal-Shabeeb and Abdulrahman Faaiq carried out the experimental work and contributed to the coordination of the experimental procedures. All authors reviewed and approved the final version of the manuscript.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

Artificial Intelligence (AI) Contribution Statement

Artificial intelligence (ChatGPT, developed by OpenAI) was used solely for language editing. All scientific content, experimental results, interpretations, and final editorial decisions were prepared, verified, and approved by the authors.

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