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Structural and Optical Properties of TIPS Pentacene Thin Film Exposed to Gamma Radiation

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This study investigates the effects of Gamma-irradiation on the structural, morphological and optical properties of 3,16-bis(tri isopropyl silylethynyl)pentacene (TIPS Pentacene) organic semiconductor films. The TIPS Pentacene thin films were irradiated at 10 to 300 kGy at a dose rate of 1.58 kGy/hr. The films were characterized using X-Ray Diffractometer (XRD), Atomic Force Microscopy (AFM) and Ultraviolet-Visible Spectroscopy (UV-Vis). The XRD analysis showed that the pre-irradiated thin films were of crystalline structure, indicating a broad wave diagram. The XRD and AFM results show that these variations can be attributed to the radiation-induced local heating and microscopic atomic mobility. Based on the UV-Vis results, the thin films exhibit approximately 70% optical transmittance in the visible region at pre-irradiation. At post-irradiation, optical transmittance decreased to 55% at the maximum absorbed dose. The corresponding optical bandgap decreased from 1.87 to 1.50 eV after a total ionizing dose of 300 kGy. The findings showed that TIPS Pentacene thin film has good mitigation towards gamma irradiation and can withstand harsh radiation while retaining its semiconductor properties. It is a potential candidate for flexible electronics for space applications.

Keywords: *TIPS Pentacene, gamma radiation, radiation effects, organic semiconductor.*

1. Introduction

Many flexible electronics devices have recently employed organic materials as the active component[1]. Hence studies on new semiconductor materials such as II-VI compound materials, two-dimensional materials, complex and simple oxides, small molecules and polymers, biological molecules, colloidal quantum dots, and perovskites have gained wide attention. These semiconductors are less expensive than their conventional inorganic counterparts that can be applied to many forms such as flexible electronics, particularly thin film transistors (TFTs) on polymer substrates, which are crucial for space exploration applications, such as space-borne telescopes, balloons solar sails and synthetic aperture radar systems[2]. These devices require distributed sensing and electronic health to monitor the thin, low mass and large area deployable structures, which cannot be implemented using conventional engineering materials such as metals and alloys[3]. However, the operating environment of these devices during storage and deployment is far from stable and has often been disturbed, especially in outer space. Hence, it becomes a major factor contributing to the components' ability to function properly in harsh radiation environments. Interaction of ionizing radiation with matter, especially gamma radiation, is crucial in both theoretical and practical[4-9]. Hence, the effect of gamma radiation on the structural, optical and electrical properties of various organic thin films and semiconductor electronics was widely investigated. Previous studies have found that when the samples are exposed to ionizing radiation, their structural, optical and electrical properties are altered[10-18]. Thus, the radiation response of the material must be thoroughly investigated before the material is exposed to a harsh radiation environment, specifically for flexible space-borne electronic applications.

TIPS Pentacene represents one of the most promising organic semiconductor materials for low-cost, flexible electronic devices due to its high solubility in common organic solvents and good environmental stability[2-5]. TIPS Pentacene is a conjugated form of pentacene commonly used for small molecule organic semiconductors due to the

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bulky functionalized group that helps increase solubilities[6-8]. TIPS Pentacene has a two-dimension π-stacking molecular structure that helps improve mobility[18]. These bulky functionalized groups assist molecular ordering in the face-to-face interface, overlapping the pi-orbital and increasing device performances[19]. Although several technological applications of TIPS Pentacene exist in microelectronics[19-22], it is quite unreliable under harsh radiation environments. In particular, the current knowledge of the radiation response of TIPS Pentacene-based devices is unclear, and the physical effects of the radiation in harsh environments are unknown[4,9]. Hence, this study investigates the physical and optical characteristics of TIPS Pentacene organic thin film upon exposure to gamma irradiation.

2. Experimental Details

The Indium Tin Oxide (ITO) substrate was rinsed using distilled water, ethanol, and acetone for 10 minutes in an ultrasonic bath. The substrate was then rinsed in distilled water and dried with nitrogen gas. The powdered TIPS Pentacene (purity > 99.5%, Sigma-Aldrich) was dissolved in toluene (purity 99.9%, Sigma-Aldrich) and stirred at room temperature for 24 hours to form a 1.0% wt solution. The organic layer of TIPS Pentacene was deposited using the spin coating method. 1 mL of TIPS Pentacene solution was dispensed and spread onto all substrate samples with the sintering of 500 rpm for 5 s. The sample was spun at a speed of 1000 rpm for 20 s.

The fabricated samples were irradiated at the MINTec-Sinagama facility, Malaysia Nuclear Agency. Using the tote irradiator model JS8900, the gamma irradiation total ionizing dose was 10 kGy, 50 kGy, 100 kGy, 150 kGy, 200 kGy, 250 kGy and 300 kGy. The activity of Co-60 was 352191 Curie with a dose rate of 1.58 kGy/hr measured by a Ceric Cerous dosimeter. As illustrated in Figure 1, lead containers accommodate samples during irradiation by gamma rays from the Co-60 source.

Using XRD, structural analysis of both the post-irradiated and unirradiated films was performed at room temperature. A MultiMode 8 atomic force microscope (AFM) was used to examine the surface properties of the samples. The samples' optical transmittance and band gap were investigated using a Lambda EZ210 UV-Vis spectrometer.

3. Results and Discussion

3.1. Structural properties of TIPS pentacene thin film

As shown in Figure 2, the structural properties of pre- and post-irradiation of TIPS Pentacene indicate the thin film maintains its crystalline properties despite rising total ionizing dose. The XRD analysis for un-irradiated thin film shows a good c-axis orientation perpendicular to the substrate surface. Two XRD spectra peaks at 5.33° and 10.25° correspond to (001) and (002) planes, respectively.

However, full-width half maxima (FWHM) exhibit a change in structure with the increase in ionizing dose. The length of peak width influences the FWHM of thin films. As the intensity becomes wider, the crystal quality of thin films deteriorates. This intensity degradation might be due to the irradiation-induced defects created in the sample[23,24].

Based on the XRD analysis, the FWHM of the thin film increases with the rising total ionizing dose, indicating the rising grain size diminished with a higher dose[25]. At the highest ionizing dose of 300 kGy, the FWHM of the irradiated TIPS Pentacene thin film is 1.08, corresponding to a grain size of 7.70 nm. The unirradiated thin film shows an FWHM of 0.11 with a grain size of 71.7 nm. The grain size can be calculated from the FWHM obtained from the XRD result by using Scherrer's equation[4,26,27]:

$$d = \frac{K\lambda}{\beta \cos\theta} \tag{1}$$

Where K, λ, θ, and β are the shape factor, X-ray wavelength, diffraction angle, and the full width half maximum (FWHM) of the diffraction peak of the thin film, respectively.

For the optical transmittance of Tips Pentacene thin film, the values are calculated using reflectivity ® and absobance(A) spectra using the following equation[28-30]:

$$T = (1-R)^2 e^{-A} \tag{2}$$

While the determination of its optical band gap adapts the utilization of Beer Lambert's Law, as in Equation 3[31]:

$$(\alpha h\upsilon)^{1/n} = \beta\left(h\upsilon - E_g\right) \tag{3}$$

Figure 1. Experimental setup inside the gamma radiation facility.

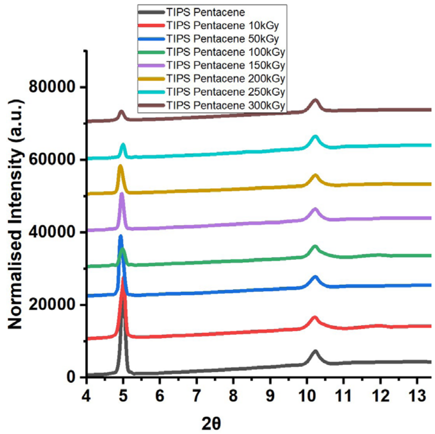

Figure 2. XRD spectra of TIPS Pentacene before and after irradiation with different total ionizing doses.

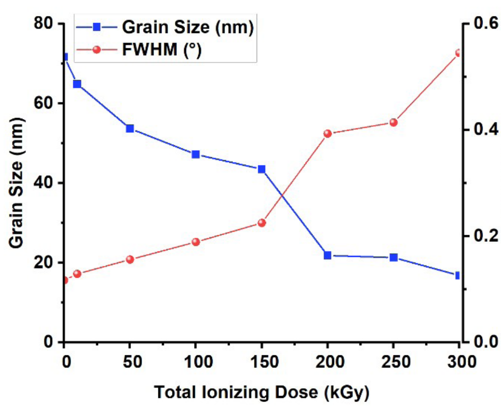

Figure 3. Variation of grain size and FWHM of TIPS Pentacene thin films with different total ionizing doses.

Where α, h, υ, and E_g represent the absorption coefficient, Planck's constant, frequency, and energy band gap, respectively, with values β as the absorption edge with parameter and n held constant as 2 for direct allowed transition.

Gamma irradiation's strong effect caused TIPS Pentacene to alter its structure, such as the widening of the FWHM, which has an inverse proportionality to the grain size. As the samples were exposed to gamma rays, the Compton interaction produced electrons that set the primary knock-on atoms in motion. The phenomenon occurs when the incident radiation gains enough energy to subsequently displace atoms within the secondary lattice structure, causing displacement cascade, structural distortion and swelling of the grain size[32,33]. Figure 3 illustrates the relationship between FWHM data obtained from the XRD analysis and the sample's grain size.

AFM was used to examine the morphological variations and surface roughness of the un-irradiated and gamma-irradiated TIPS Pentacene thin films. The results of the AFM images of the TIPS Pentacene thin film are shown in Figure 4. It is observed that the grain size decreased with a higher total ionizing dose. The reduced grain size can be attributed to the ionization of the thin films caused by gamma irradiation. The shrinking grain size increases its electrical conductivity. Thus, exposure to ionizing radiation increased the turn-on-voltage. The decrease of majority charges resulted in generating vacancies under irradiation and charge capture on defects[34].

Surface roughness significantly influences carrier mobility and carrier scattering. High surface roughness increases carrier scattering and acts as a trap state in the channel, declining the electronic device's performance[35]. Referring to Figure 4, the surface roughness decreased from 77.03 nm for an unirradiated thin film to 29.65 nm for samples exposed at the highest ionizing dose of 300 kGy. Concerning the modification of the surface topology, the degradation with a higher total ionizing dose is proven through particle analysis, where the average density of TIPS

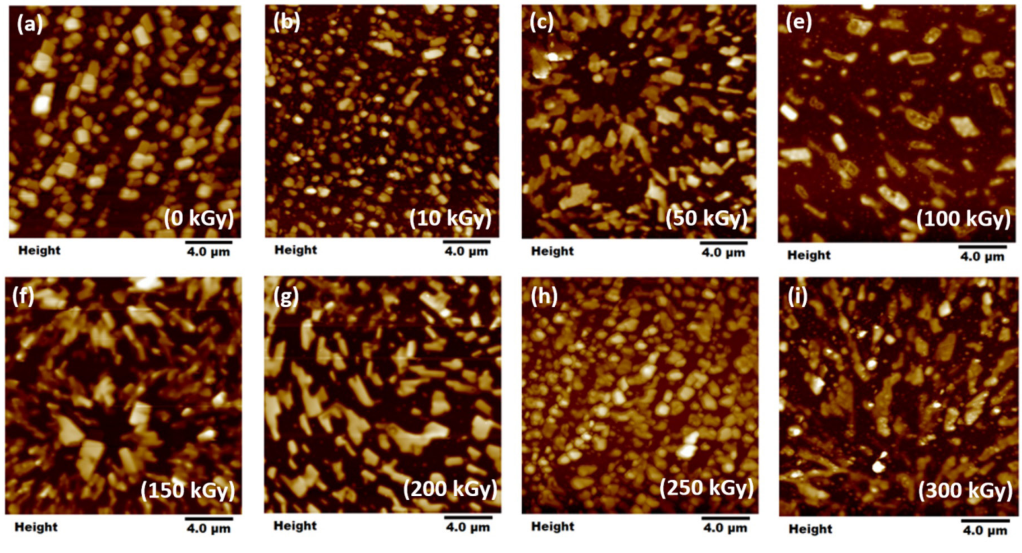

Figure 4. 2D AFM images of un-irradiated and gamma irradiated TIPS Pentacene thin films.

Pentacene decreases from 0.55 μm^{-2} to 0.215 μm^{-2}with a higher dose of radiation. The results justify the decrease in grain size drops with a higher total ionizing dose attributed to the reduction in the overall density. However, the results may very well be attributed to homogeneity differences within the surface of the scanned area.

As indicated in Table 1, it is observed that a higher ionizing dose causes a decrease in grain size, RMS roughness and particle density of all films. The degradation of TIPS Pentacene shows a strong breakdown in its material properties with higher radiation exposure[36,37]. Despite the grain quality degradation, organic-based semiconductors' performance cannot be applied directly to their electrical performance due to its molecular properties[38].

3.2. Optical properties of TIPS pentacene thin film

Figure 5 shows the absorbance and transmittance of irradiated and un-irradiated TIPS Pentacene thin films for wavelengths from 300 to 1100 nm. Overall, the transmittance of all thin films decreased after gamma irradiation. It decreased from 70% to 55% in the wavelength region of 380 to 700 nm[39]. The reduction in transmittance with a higher dose is due to the surface and lattice defects produced via radiation (voids and pores)[9]. The higher vacancy defects simultaneously increase its absorbance.

Table 2 shows the optical bandgap values of TIPS Pentacene for different gamma-absorbed doses. The decrease in bandgap is due to the lattice defects produced by radiation exposure, causing structural and morphological changes[40], as shown in Figure 3. Furthermore, the decrease in the band gap energy is due to the increase in crystallinity, as validated by the XRD measurements. The irradiation could initiate new energy levels in the forbidden gap, causing radical changes in the carrier concentration in the material.

Figure 6 shows the refractive index dispersion for un-irradiated and irradiated TIPS Pentacene thin films. It is observed that the refractive index of the investigated films decreases with higher gamma radiation dose. The results are due to the decreased density of the investigated films with irradiation due to ionization from gamma rays' collision with the thin film, altering its structural properties[41]. Figure 7 shows several optical conductivity spectra for differing gamma-absorbed doses. It is observed that the optical conductivity of the thin films is shifted to the lower energy.

Based on the optical transmittance, the optical band gap of TIPS Pentacene thin films was calculated using the intercept of the Tauc plot[42-44]. TIPS Pentacene thin film's common band gap value is 1.72 eV[45,46]. The optical band gap observed for an unirradiated sample and at maximum ionizing dose is 1.87 eV and 1.50 eV, respectively. The decrease in band gap at the irradiated film is caused by a slight 'red shift' in the optical spectra and the rising energy width of the band tails of localized states. Structural defects due to radiation effects also influence polarization and spontaneous polarization along the c-axis and a-axis of the local electric

Table 1. Summary of structural parameters of TIPS Pentacene thin films before and after irradiation with different total ionizing doses.

Total Ionizing Dose (kGy)	RMS Roughness (nm)	Particle Density (μm^{-2})
0	77.03	0.550
10	76.44	0.072
50	59.23	0.192
100	58.37	0.213
150	54.72	0.200
200	40.72	0.095
250	32.14	0.108
300	29.65	0.215

Table 2. Optical bandgap of TIPS Pentacene thin film.

Total Ionizing Dose (kGy)	Optical Bandgap (eV)
0	1.87
10	1.81
50	1.75
100	1.71
150	1.68
200	1.62
250	1.59
300	1.50

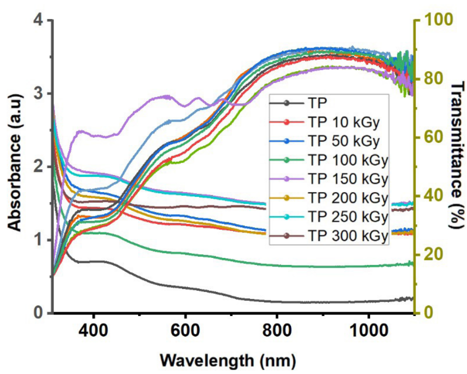

Figure 5. Transmittance and absorbance spectra of different gamma-absorbed doses of TIPS Pentacene.

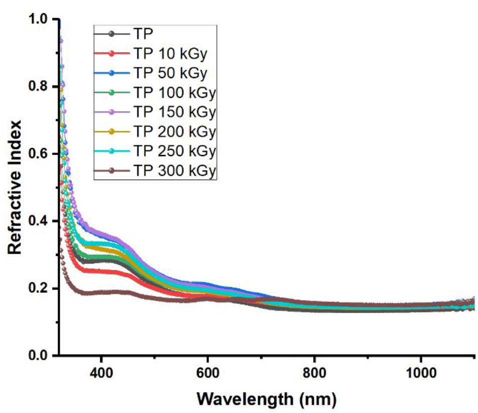

Figure 6. Refractive index of different gamma-absorbed doses of TIPS Pentacene.

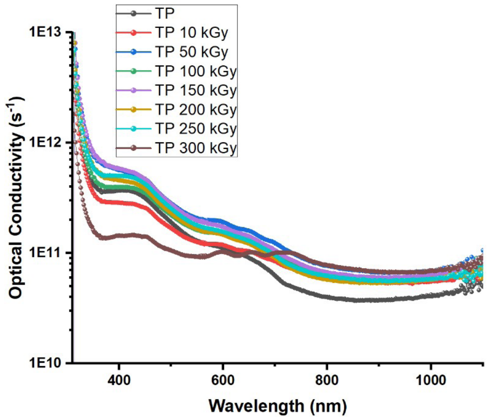

Figure 7. Optical conductivity of different gamma-absorbed doses of TIPS Pentacene.

fields. The defects can lead to band bending at the crystallite boundaries, affecting energy band gap, increasing the conductivity of irradiated thin films[47]. This finding is in agreement with other previous studies[9,24,34,42].

4. Conclusion

The results of the irradiated samples demonstrate the benefits of modifications in their structural and optical properties compared to the unirradiated sample. XRD analysis showed that unirradiated and irradiated TIPS Pentacene thin films have crystalline structures, maintaining their semiconductor properties. The gamma-irradiated film exhibit higher aggregation on its surface morphology than the unirradiated film. The surface analysis by AFM also indicated that the grain size decreased after gamma-irradiation, which was validated using Scherrer's equation. The transmittance also decreased with the higher dose. The optical band gap decreases from 1.87 eV for the un-irradiated samples to 1.50 eV at the maximum total ionizing dose of 300 kGy. These structural and optical parameters alterations by gamma-irradiation make these thin films a promising material to replace conventional silicon-based semiconductor materials for the integrated optical device in terrestrial applications.

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Erratum

In the article “Structural and Optical Properties of TIPS Pentacene Thin Film Exposed to Gamma Radiation”, with DOI: https://doi.org/10.1590/1980-5373-MR-2022-0227, published in Materials Research, 25:e20220227, in the “Acknowledgments” section, where it was written:

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Should read:

The authors wish to acknowledge the support of the Ministry of Higher Education for providing funding assistance based on the Fundamental Research Grant Scheme FRGS/1/2020/STG07/UMS/02/1 titled “Explication On The Damage Mechanism Induced By High Energy Radiation On ZnO Based Photoconductive Radiation Detector For Space Borne Application”, and GUG0387-2/2019, with the title “Investigation of Damage Mechanism of Hybrid ZnO based Devices due to Ionizing Radiation for Flexible Electronic in Space Application”.