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Defect characteristic of oxygen-deficient Ge-doped preform using photoluminescence spectroscopy

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ABSTRACT

Study has been made of the Photoluminescence properties of Ge-doped silica preforms fabricated using the MCVD process and subsequently subjected to γ-ray irradiation. The photoluminescence emissions pointed to the presence of defects related to oxygen vacancies. Two types of preform were fabricated, obtained using a different flow rate and deposition temperature for each case. Results from the absorption spectra of the samples named as P1 and P2, show a signature absorption peak at 5.1 eV and 6.8 eV, indicative of oxygen-deficient and oxygen-rich defects respectively. Photoluminescence investigation have been carried out before and after the irradiation process with both samples reveal two main peaks at 1.5 eV. The highest intensity at 1.5 eV is known as an interaction between the Non- Bridging Oxygen Hole Centre (NBHOC) with the presence of impurity in the glass matrix. Upon irradiation, weak peak can be observed at 1.8 eV, sample P1 and P2, the PL intensity increases by a factor of 20 × and 50 × , respectively. This peak is associated with the oxygen deficient state in the sample. The peak referring to defect known as Germanium Lone Pair Centre (GLPC) are observed in both samples, peak shown at 3.1 eV, regardless of the Germanium Oxygen Deficient Centre (GODC) content. In regard to this, it can be concluded that this defect is generated independently in all germanium samples and are not correlated with the GODC band observed in the absorption band.

1. Introduction

Defects present in the silica network gives rise to optical absorption and luminescence. Defects can occur under certain influencing conditions in the fabrication process, including precursor flow rate, temperature, and the glass drawing processes. Several workers have studied the conditions for such defects to occur, examining for instance dependence on the oxidation environment during fabrication (Fujimaki et al., 1997; Skuja et al., 2005). Particular examples of the defects that are determined by the oxidation environment are oxygen-rich and oxygen-deficient defects. Pure or doped silica preform with defects states can show both associated optical properties, probed by for example Raman, UV–Vis and photoluminescence spectroscopy. These underpin the performance properties of such silica media when applied as radiation dosimeters (Anedda et al., 2001; Tamchek et al., 2013).

One of the main desirable properties of germanium is the photosensitivity, a matter of importance in the process of UV writing of refractive index gratings in optical fibers. Much attention is being paid to the defect produced by the photosensitivity, known as the germanium oxygen deficient centre (GODC). This defect is commonly considered to be responsible for the changes in the refractive index in germanosilicate glass (Dianov et al. n.d.; Heaney and Erdogan, 2000). Concerning the absorption optical activity of these germanium related defects, it has been observed to have connection with the band peaks at 240 nm and 245 nm, referred to as the GODC and NOV (neutral oxygen vacancy) respectively (Hosono et al., 1992a; Kohketsu et al., 1989). These two

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defects have sometimes been assigned to wrong bonds, such as Ge–Ge, Si–Ge, rather than the usual Si–O-Ge bond in Ge-doped-silica media. It is found that these defects can be bleached with UV irradiation to produce other defects such as GeE', Ge (1) and Ge (2).

The Germanium Lone Pair Centre GLPC defect centre arises due to the presence of lone pair electrons on the germanium atom within the crystal lattice. These lone pair electrons can function as a charge carrier trap for electrons or holes, which may reduce the material's conductivity. Furthermore, the existence of GLPCs can influence the optical characteristics of the material. When the lone pair electrons are energized to higher states, they can interact with the valence or conduction bands of the material, leading to the formation of defects that can produce light. GLPC has been illustrated to be similar to the Ge (2) defect. This defect can be observed in two band peaks in the photoluminescence spectrum, at 3.2 eV and 4.3 eV, related to the transitions from the excited electronic states of singlet (S1) and triplet (T1), respectively, to the ground state (S0) (Skuja, 1992). This defect is related to the absorption band at 5.1 eV with optical transitions from the ground singlet state (S0) to the first single state (S1) (Hosono et al., 1992b).

The GeE' is illustrated as (≡Ge•), associated with an absorption band at 6.2–6.4 eV. The Ge (1) defect consists of an electron trapped at the site of a substitutional 4-fold coordinated Ge precursor (GeO•4), attributed to the absorption band at 4.4–4.6 eV (Chiodini et al., 1999; Neustruev, 1994; Pacchioni and Mazzeo, 2000). The Ge (2) defect is assigned as ionized twofold coordinated Ge (=Ge••) (Skuja, 1992). Based on EPR analysis, the presence of GeE, Ge (1) and Ge (2) are found to be in the region g = 1.9937, 1.9933 and 1.9866 respectively (Nishii et al., 1999).

For this reason, in present work the parameters for fabrication of the preform have been made to vary in order to create an oxygen deficient and oxygen rich environment in the preform. In Oxygen-deficient Ge-doped preforms are known to exhibit certain defects that can impact the performance of the resulting fibre. Understanding the characteristics and mechanisms of these defects is crucial for improving the quality and reliability of the fibre. To obtain a more complete evaluation of the defects, Photoluminescence investigation have been carried out before and after the irradiation process. The existence of key spectroscopic peaks after and before irradiation have then been analyzed, study focusing on the defects induced by ionizing radiation.

2. Methodology

2.1. Fabrication of oxygen deficient preform

In this paper, we fabricated two types of preform labelled as P1 and P2. The fabrication of this two preforms has been reported in Siti Shafiqah et al. (2015). The Ge-doped preforms are synthesized using the MCVD technique with $SiCl_4$ and $GeCl_4$ as the main precursors. The oxygen flow rate applied is shown in Table 1, being made to vary to produce the oxygen-deficient and oxygen-rich preforms. The process temperature and annealing (collapse) duration are also made to vary, allowing study of the effect of oxidation and diffusion in preforms. The process temperature and annealing (collapse) duration have also been made to vary, allowing study of the effect of oxidation and diffusion in the preforms. Table 1 shows an example of the resulting preform used in this present work.

Table 1
Precursor flow rate and deposition temperature.

Parameter	P1	P2
$SiCl_4$ (sccm)	100	100
$GeCl_4$ (sccm)	200	150
Oxygen (sccm)	1400	1600
Temperature (°C)	2100	2200
Collapse speed (mm/min)	2	4

*Note: It is typical for the precursor flow rate to be measured in standard cubic centimetres per minute (sccm).

2.2. Gamma irradiation

In regard to the irradiations made during this series of investigations, all samples were exposed to gamma irradiation, delivered by a University of Malaya Gammacell model 220 ^{60}Co source. All samples were irradiated in air, the calculated dose-rate being 0.0469 Gy/s. The duration of irradiation for a delivered doses of 10 Gy has been obtained on this basis.

2.3. Photoluminescence

Photoluminescence (PL) spectroscopy carries useful information that can facilitate sample analysis and improve and augment data obtained from Raman measurements. PL involves both fluorescence and phosphorescence processes and originates from an absorption/emission process between different electronic energy levels in the material. In present study, the use of PL spectroscopy is to detect the presence of defects related to oxygen vacancies for instance NBOC, thus supporting the result collected from absorption spectroscopy. The system with a 40× objective lens lases at 325 nm, providing for target excitation. The grating system offers 1200 lines/mm and the PL intensity is detected using a CCD camera of 578 × 400 pixels.

3. Results and discussion

3.1. Optical absorption

Fig. 1 shows the absorption spectra of the Ge-doped silica preforms has been reported by Shafiqah et al., (2015). The absorption coefficients are obtained by dividing the absorption value with the sample thickness. Two main peaks can clearly be observed, at 5.1 eV and 6.8 eV. The larger absorption peak at 5.1 eV which is apparent only in P2, is assigned to the germanium-oxygen deficient centre (GODC) while the combination of unresolved peaks at 5.06 eV and 5.16 eV are due to the neutral oxygen vacancy (NOV) and the germanium lone-pair centre (GLPC), respectively. The absorption activity at 5.1 eV for both pure and Ge-doped silica grown in an oxygen deficient condition has been reported by a number of workers (Skuja, 1992; Yuen, 1982). It has been claimed elsewhere that in oxygen rich samples, a peak at 5.1 eV is not observed (Anedda et al., 2001; Fujimaki et al., 1997; Tohmon et al., 1989a). Thus, the indication is that P2 is in an oxygen deficient state while P1 is in an oxygen rich state.

3.2. Photoluminescence

Photoluminescence (PL) spectroscopy studies were carried out before and after γ-irradiation to a dose of 10 Gy, use being made of a laser providing excitation at a wavelength of 325 nm from the He–Cd laser line. All spectra were normalized to the highest intensity wavelength, at a commensurate energy around 1.5 eV. The results obtained for P1 and P2 are presented in Fig. 2 (a) and (b), respectively. Both samples reveal two main peaks at 1.5 eV and 3.1 eV. The PL spectra consists of a broad band peak rather than sharp lines, reported to be due to electron-phonon coupling (Linards Skuja, 1998). The highest intensity at 1.5 eV is similar to the findings of (Dragic et al., 2008), explained by the authors as an interaction between the Non- Bridging Oxygen Hole Centre (NBHOC) with the presence of impurity in the glass matrix. This is further supported by the findings of (Das et al., 2012), the authors reporting the peak at 1.67 eV to be correlated with the effect of Ge nanocrystals with the host matrix. Further analysis has been carried out within the region 1.7–2.1 eV, as here we can observe increases in the PL intensity.

Fig. 3 (a) and (b) for P1 and P2 respectively, show analysis around a weak peak at 1.8 eV, showing the intensity after irradiation to be

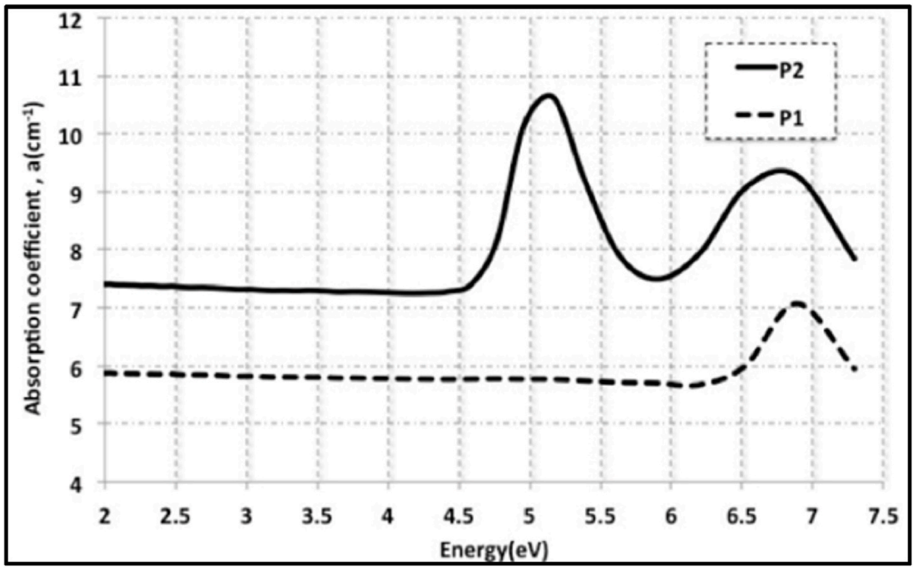

Fig. 1. Absorption spectra for P1 and P2. Two main peaks can be observed, at 5.1 eV and 6.7 eV. The peak assigned to GODC at 5.1 eV is only apparent in P2 (Shafiqah et al., 2015).

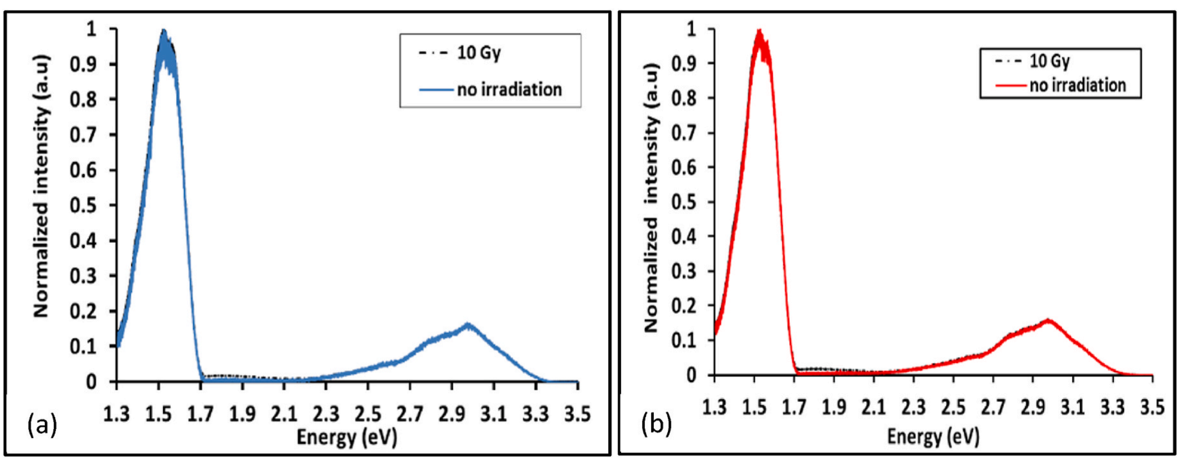

Fig. 2. PL spectra of (a) P1 and (b) P2 in condition of before and after 10 Gy of γ-irradiation.

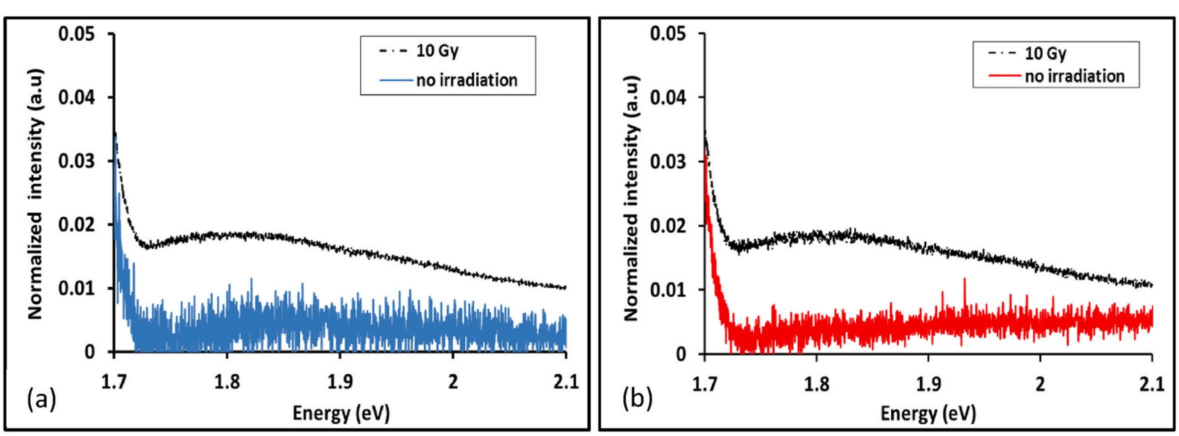

Fig. 3. PL spectra for P1 and P2 in energy region of 1.7–2.1 eV.

significantly greater than before irradiation, suggesting the defect centre at this peak to be enhanced by the irradiation. The origin of this 1.8 eV band has been ascribed to the NBHOC in pure silica glass irradiated with ionizing radiations or ultraviolet lasers (Awazu and Kawazoe, 1990; Sigel and Marrone, 1981; Tohmon et al., 1989b). NBHOC in pure silica is presented as ≡Si – O•, where the symbol ≡ represents bonding to three oxygens and the symbol • represents an unpaired electron which is a dangling bond. Further, with both samples showing almost equal PL amplitude before and after the irradiation process, this is potentially indicative of the NBHOC defect showing insignificant dependency upon the type of Ge-doped silica samples; this will be explored in more detail below. NBOHC, a dangling bond type defect, had not previously been reported to have been observed in quartz monocrystals irradiated by γ-rays under conditions in which only single oxygen vacancies could be created by fast secondary electrons (Skuja, 1998).

Sakurai et al. (1999), in a paper based on unnormalized PL spectra of silica glass, ascribed the 1.8 eV peak to be associated with the oxygen deficient state. They observed an increase in terms of the PL intensity with decrease in the amount of oxygen in the sample (Sakurai et al., 1999). We have thus applied the technique of Sakurai et al. the results being presented in Fig. 4. The findings are similar to those of Sakurai et al. With sample P2 irradiated up to a dose of 10 Gy, the PL intensity increases by a factor of 50 × compared with the unirradiated sample. This indicates enhancement of the 1.8 eV peak in oxygen deficient state samples, a result of the action of γ-irradiation. Sample P1 reveals a similar peak with increase of PL intensity by a factor of 20 × . This can be explained in terms in the formation of the oxygen deficient state and the action of γ-irradiation. Under γ-irradiation, oxygen deficient states are formed in P1, due to the displacement of oxygen from the Si–O–Si bond, as the result of the nonradioactive decay of self-trapped excitons (Itoh et al., 1990). The formations are expressed in equation (1).

$$\mathrm{Si-O-Si \rightarrow Si-Si + O\ (interstitial)} \quad (1)$$

By comparing the above we suggest that our findings, concerning the 1.8 eV peak, to be associated with the oxygen deficient state in the sample, similar to the findings of Sakurai et al. (1999).

Deconvolution analysis in this region is presented in Fig. 5, the highest peak observed being used as the reference. Comparison of the results of the two samples show the peak energy and FHWM to be slightly different. With decrease in the amount of oxygen in the sample (from P1 to P2), the peak is observed to shift to lower energy, from 1.86 to 1.84 eV while the value of FWHM decreases by around 1.3%. This is due to difference in the presence of PL centres associated with the oxygen deficient state introduced during sample preparation.

The peak at 3.1 eV (see Fig. 2) is assigned to the Germanium Lone-Pair Centre (GLPC), typically present in most Ge-doped silica fiber (Awazu et al., 1990; Linards Skuja, 1992). The electronic transition for the GLPC at 3.1 eV is ascribed as T_1 to S_O. This defect has been correlated with the diamagnetic Ge oxygen deficient centre (GODC) and in terms of microscopic structure is twofold coordinated Ge (= Ge ··), where = indicates bonding with two oxygen atoms, while ·· indicates an electron lone pair; from this structural arrangement, the defects have been accorded the name of Ge lone-pair centre (GLPC). Wu et al. (1999) have correlated this peak with an absorption optical band at 5.1 eV which refers to the GODC; see also our absorption spectrum for P2 in Fig. 1, recording our own observation of the 5.1 eV peak in an oxygen deficient sample (Wu et al., 1999). Based on our results, it is interesting to note here that the GLPC creation are observed in both samples, regardless of the GODC content. In regard to this, it can be concluded that this defect is generated independently in all germanium samples and are not correlated with the GODC band observed in the absorption band. Alessi et al. (2008) has shown results that are in agreement with ours, showing evidence that the GLPC are observed in both samples, regardless of the peak at 5.1 eV. Further investigations of this band concern the intensity recorded before and after the γ-irradiation, use being made of the deconvolution technique. From Figs. 6 and 7, the broad peak in this region consists of five deconvoluted peaks, located around 2.6, 2.8, 2.9, 3.0, and 3.1 eV, with the highest energy peak recorded at 3.0 eV. In the energy range 2.5–3.1 eV, several researchers have reported the present peaks to be attributable to oxygen vacancy pairs and related defects. Although most have reported the GLPC to arise at 3.1 eV, we suggest our results to have shifted to lower energy, to 3.0 eV. This is because this peak is known to shift to lower energy with decreasing value of excitation energy (Alessi et al., 2008). In both preform samples, the intensity at this band peak points (Table 2) to an increase following irradiation. This peak shows similar behavior to that at 1.8 eV, suggesting that γ-irradiation enhances the GLPC defects. Alessi et al. (2008) has also reported on high purity a-SiO_2 (*a* indicating the amorphous state), it being found via ion implantation or gamma irradiation that the photosensitive two-fold coordinated Si(=Si··) can be induced, featuring an absorption band at 5.06 eV and two related emissions at ~ 4.4 eV and ~2.7 eV. The 5.06 eV peak, assigned to the main generation mechanism of twofold coordinated Si defects, is suggested to involve the displacing of O from bonding configuration due to knock-on or radiolytic processes. However, this peak does not appear in any of our results.

4. Conclusion

Absorption analysis showed the P2 preform to be characterized by the presence of a peak at 5.1 eV attributed to the GODC, a result obtained for both pure and Ge-doped samples grown in an oxygen deficient environment. The peak at 5.1 eV is not apparent in P1, indicating that the P2 preform is oxygen deficient and P1 is oxygen rich. From PL analysis of the Ge-doped preform, the peak at 1.8 eV has also been found to be associated with the oxygen deficient state in the sample. The PL intensity of P1 and P2 increased by a factor of 20 × and 50 × respectively compared with the un-irradiated samples. For the Ge-doped media, the peak at 3.1 eV, which has been reported by others and assigned to the GLPC (Ge Lone-Pair Centre), has also been found to be present in both of the Ge-doped precursor samples. Results from present

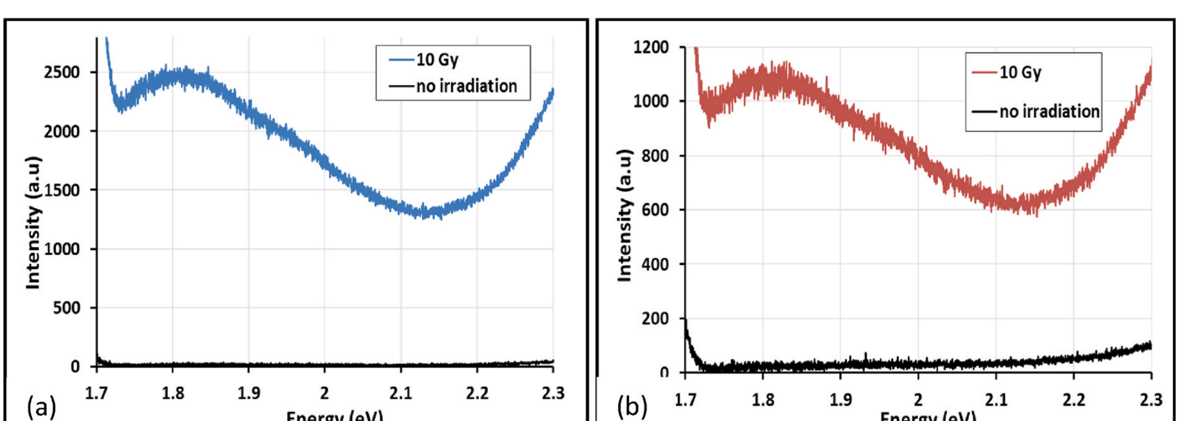

Fig. 4. PL spectra for (a) P1 and (b) P2 on unnormalized data, in the region 1.7–2.3 eV.

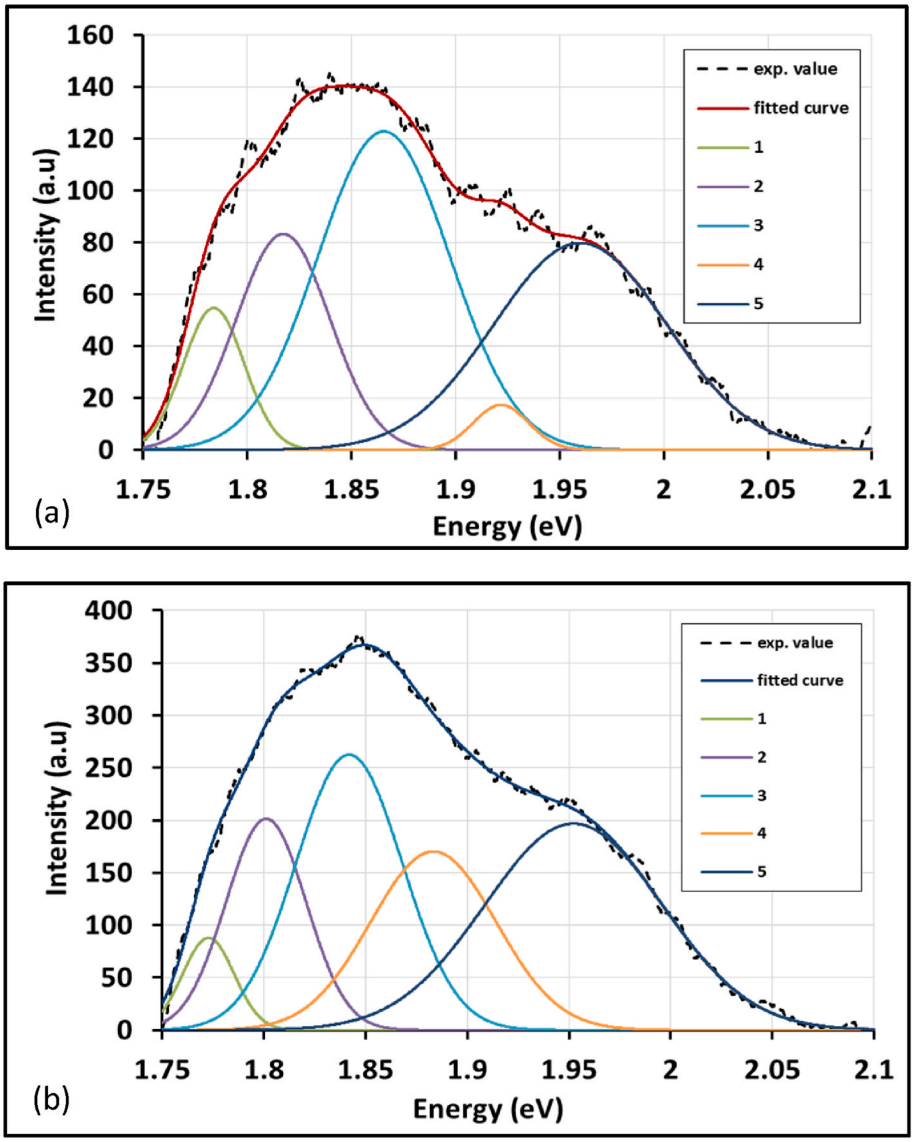

Fig. 5. Deconvolution of the experimental curve at peak 1.8 eV of sample (a) P1 and (b) P2 following γ-irradiation.

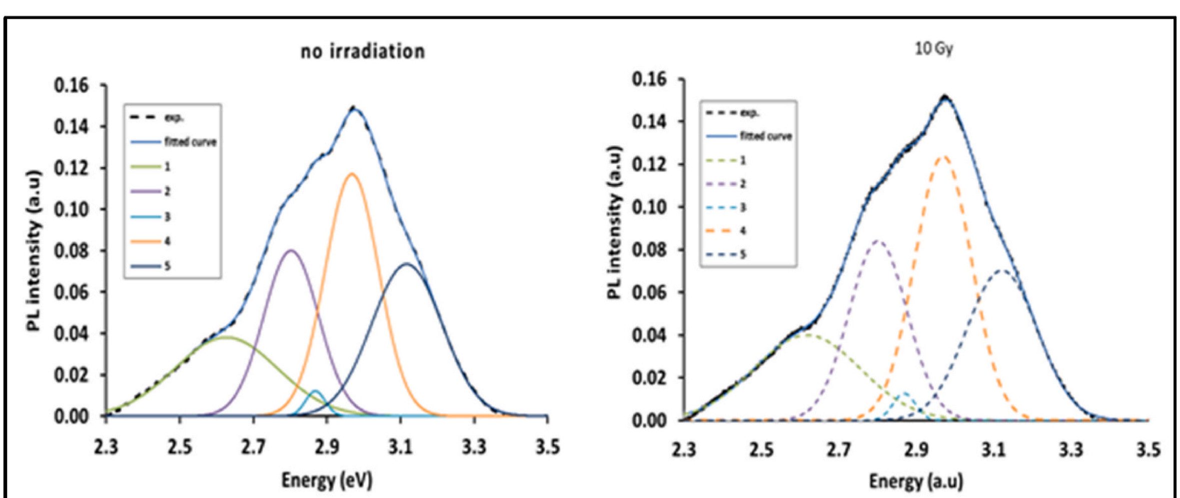

Fig. 6. Deconvolution of the PL peak for sample P1.

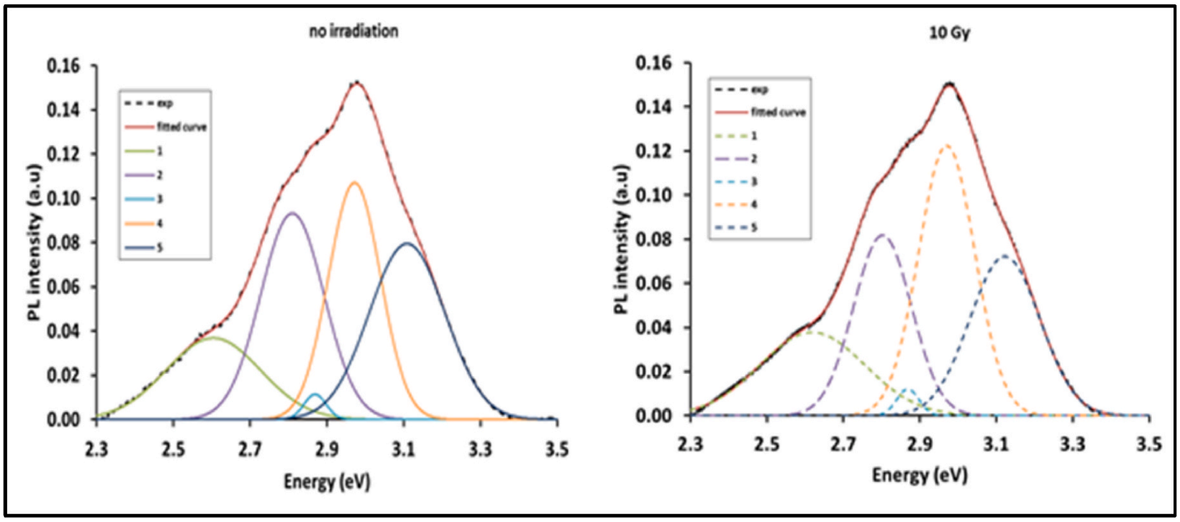

Fig. 7. Deconvolution of the PL peak for sample P2.

Table 2
Deconvolution analysis at the PL peak 3.0 eV (the highest peak observed in the deconvolution analysis).

Sample	Before irradiation		After irradiation	
	Intensity (a.u.)	FWHM (eV)	Intensity (a.u.)	FWHM (eV)
P1	0.117	0.171	0.124	0.173
P2	0.107	0.122	0.123	0.173

work have lead to the conclusion that this defect is generated independently and has no correlation with the GODC band peak observed in the absorption analysis.

Author statements

This study was conducted by A.S. Siti Shafiqah and analyzed the data with S.F. Abdul Sani and Nizam Tamchek. K.S. Almugren and F.H. Alkallas provide contributions to the research consultation and financial support. D.A. Bradley provides valuable input on radiation physics and contributed to the writing and scientific editing of the manuscript.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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