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The Use of Polycarbonate Film in High-Level Electron and Gamma Dosimetry

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Dosimetrical properties of polycarbonate film for high-level dosimetry of electrons and ^{60}Co gamma-rays have been examined. Polycarbonate film of 0.1 mm in thickness was chosen as the most suitable film for this purpose. It can cover the dose range 1.0 - 300 Mrad. The amount of scatter in measured value is within 3.5%.

The radiation induced optical density at 330 nm shows rapid fading of 7 - 13% for the initial one day after irradiation at room temperature and subsequent fading rate is very small, about 0.4% per day. The fading depends on the absorbed dose, storage temperature, film thickness and wavelength. The effects of the storage time and temperature after irradiation and the irradiation temperature in this film are presented.

For practical dosimetry, it is necessary to stabilize the induced optical density by storing the irradiated film for a day or by heat treatment at 100°C for an hour.

The usefulness of polycarbonate film in high-level dosimetry is discussed compared with poly(ethylene terephthalate) film dosimeter and cellulose triacetate film dosimeter.

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電子線および γ 線の大線量測定における ポリカーボネートフィルムの利用

日本原子力研究所高崎研究所開発試験場

田中隆一・劉　永秀*・田村直幸

（1977年2月3日受理）

ポリカーボネートフィルムの吸光度変化を利用する電子線およびコバルト γ 線の大線量測定の試験を行なった。0.1mm の厚さのフィルムが最適な試料として選ばれた。測定可能な線量範囲は 1～300 Mrad であり，試料間のレスポンスのバラツキは 3.5％以内であった。

放射線によって誘起された 330 mm における吸光度は試料を照射後1日間室温で放置することによって 7～13％のフェーディングを示すが，その後のフェーディングは少く，1日あたり約 0.4％である。フェーディングは吸収線量，保存温度，フィルムの厚さ，波長に依存する。照射後の保存時間や温度の効果，照射温度の効果について述べる。

実用的な測定のためには，照射されたフィルムを1日間保存するかあるいは 100℃で1時間熱処理することによって吸光度を安定化する必要がある。

また，大線量測定におけるポリカーボネートフィルムの有用性についてポリエチレンテレフタレートフィルム線量計および三酢酸セルロースフィルム線量計との比較検討を行なった。

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* 韓国原子力研究所

CONTENTS

1. Introduction

Many transparent plastics have been developed for the radiation dose measurement in the megarad range: they are poly(vinyl chloride),[1-2] poly(methyl methacrylate) (PMMA),[3),4] cellulose triacetate (CTA),[5] poly(ethylene terephthalate)(PET),[6),7] etc. Some of them are actually used as reliable dosimeters in the fields of radiosterilization and radiation chemistry. In these fields a practical dosimeter was required for the measurement in the range of 1 - 100 Mrad related to the increase in large radiation sources.

Recently, polycarbonate film has been examined for the use in the field of high dose measurement. A simple dosimeter using polycarbonate film has been designed by Douglas[8] et al. for the measurement of dose from 0.1 to 100 Mrad of ^{60}Co gamma-rays and also Endo[9] reported the use of thin polycarbonate film as a dosimeter covering a range from 1 Mrad up to 100 Mrad for electrons. Unirradiated polycarbonate film is transparent to visible light. After irradiation it shows an absorption in the ultraviolet range; the optical density is related to the amount of absorbed dose in the appropriate range. This paper outlines in detail the dosimetric characteristics of polycarbonate considering the need for a cheap, accurate, simple, and reliable secondary dosimeter in the megarad range. The experiments have been performed with electron beam and ^{60}Co gamma-rays. The effects of the dose and the storage time and temperature after irradiation have been investigated.

2. Experimental

2.1 Material

Polycarbonate film used is Teijin Pan Light Film; the film thickness is 0.05 mm, 0.1 mm and 0.2 mm and the density is 1.2 g/cm^3. The uniformity of the film thickness used in the present work is approximately ±1.5 %. All samples were cut to a standard size 4.5 cm × 1.2 cm for convenience in the spectrophotometric measurements. The films were washed in ethanol detergent solution and wiped with soft tissue paper and sealed in a black plastic envelope to protect it from scratches and light before and after use.

2.2 Irradiation

Irradiation of the polycarbonate film was performed with an electron accelerator and a ^{60}Co irradiation facility. The electron irradiation was carried out using a Cockcroft-Walton type electron accelerator at a

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conveyor speed of 3.1 m/min, the energy was 1.5 MeV, and the beam current 3 mA. Under these conditions the absorbed dose was 1.67 Mrad per pass and the average dose rate in the center of the scanner was 5.17×10^5 rad/sec. All irradiations were performed at the center of the scanned beam. The absorbed dose was determined by a graphite calorimeter.[10),11)]

The gamma-ray irradiation of dosimeter was carried out by means of a 5×10^4 Ci ^{60}Co slab source. Irradiations were performed at the exposure rate of 2×10^6, 1×10^6 and 4×10^5 R/h at room temperature. Dosimeter films were sandwiched between Perspex sheets 3 mm thick to obtain electronic equilibrium and hold up by the aluminum supporter for irradiation. The exposure rate was determined by a parallel-plate iqnization chamber[12)] calibrated with a standard ionization chamber in low dose-rate region.

2.3 Optical density measurement

The ultraviolet absorption was measured with Japan Spectroscopic Model UVIDEC-2 digital spectrophotometer at a wavelength range from 285 nm to 500 nm. In most cases complete spectra were plotted for each film and some of them were detected at some selected wavelengths. Since the transparent polycarbonate film absorbs ultraviolet light before irradiation, the pre-irradiation optical density of each film must be subtracted from the optical density after irradiation. Since the values of initial optical density are scattered, the initial optical density of each sample should be measured in each run. And the samples which have too large initial optical density were rejected from the specimens.

3. Results and Discussion

3.1 Absorption spectrum

Typical absorption spectra for irradiated and unirradiated polycharbonate samples 0.1 mm thick are shown in Fig. 1. The curve C is radiation-induced spectrum obtained by subtracting A (unirradiated) from B (irradiated). The opitcal density of unirradiated sample rapidly decreases with increasing wavelength until 350 nm, and above this wavelength it decreases slowly. On the other hand, the absorption spectrum of irradiated sample has two shoulder peaks around about 300 nm and 400 nm.

In order to select a suitable wavelength and film thickness for the dosimeter, the effects of dose and film thickness on the radiation-induced absorption curves were examined. Figure 2 shows typical absorption spectra

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for the samples of different film thickness. The plateau around 300 nm or 400 nm is more clearly observed for thicker samples.

The absorption spectra of the sample 0.1 mm thick are shown in Fig. 3 for three different doses. All samples substantially exhibit the similar spectral changes, but the plateau around 400 nm is most clearly observed at the lowest dose. Samples irradiated with ^{60}Co gamma-rays showed similar spectra in this wavelength region. It may be said that radiation-induced spectra do not depend on dose rate.

3.2 Fading of the induced absorption

The optical density of plycarbonate film induced by irradiation is unstable like many other plastics dosimeters and it fades with time after irradiation. Effects of absorbed dose, film thickness, wavelength, and storage conditions on the fading were investigated. The changes of the spectra were observed after various intervals. The irradiated films were stored in dark at room temperature for a period of two months or more.

As shown in Fig. 4 the shoulder around 400 nm rapidly vanished with the elapsed time after irradiation. This corresponds to Barker's result[13] that there is an active center near 400 nm which vanishes with diffusion of oxygen into the sample. A minimum change was observed around 330 - 340 nm. Since the spectrum after 7 days storage did not almost further change, this curve seems to be a final shape for radiation induced spectra.

Relative changes of optical density with time after irradiation for films 0.1 mm thick are shown in Fig. 5 for the selected wavelengths. Radiation induced optical density decreases rapidly during the first one day after irradiation for samples kept at room temperature, and subsequent changes are very slow say less than 0.5 % per day at 330 nm. The decrease during the first one day at 330 nm is about 10 - 16 % of the initial optical density. Therefore, a storage of the sample after irradiation is necessary to reduce dosimetric error due to fading. The dependence of the fading on the absorbed dose was also observed. The fading at the lower dose is greater than that at the higher one. Fading observed at 330 nm is the smallest of all selected wavelengths.

Figure 6 shows fading characteristics over a period of 7 h at room temperature for films 0.2, 0.1 and 0.05 mm thick irradiated to 30 Mrad. The film 0.2 mm thick shows a significant fading at longer wavelength, while 0.1 and 0.05 mm samples show small decrease compared with the film 0.2 mm thick. The largest drop in the optical density is shown in the film 0.2 mm thick at

the wavelength of 350 nm, amounting to about 17 % after 7 h storage. Fading rate for the film 0.1 mm thick irradiated with gamma-rays is about 7 % at 330 nm after 6 days storage at room temperature, and the corresponding decrease for electron irradiation is about 12 %. This difference is probably due to dose rate or irradiation time.

The long-term fading over a period of three months for the 0.1 mm thick film at room temperature are shown in Fig. 7. The fading occurs within the first one week after irradiation, and thereafter the optical density becomes nearly constant.

If all measurements are made at 330 nm, very little fading takes place even up to 3 months after irradiation. In this point the choice of 330 nm as the measurement wavelength is advantageous.

The effect of keeping the irradiated samples in vacuum, 0.01 mmHg, at room temperature is also shown in Fig. 7. It is found that fading is not critically affected by oxygen contents of surrounding atmospehre. This is advantageous in dosimetry in the case of long time irradiation in which generally oxygen effect is a serious problem.

3.3 Effects of storage temperatures and irradiation temperatures

The fading of optical density in irradiated polycarbonate film depends on the storage temperature. Figure 8 shows the effect of storage temperature on the fading in the film 0.1 mm thick; the samples were kept at 5, 50, 75 and 100°C after electron irradiation to 30 Mrad. It is evident that the fading rate is higher at higher temperatures. At 100°C the optical density decreases within an hour and reaches almost a stable value. The decrease of optical density after six days storage at 100°C is 10 - 22 % of its initial value depending on the wavelength, while the decrease at 5°C is only 6 - 8 %.

Figure 9 shows the long-time fading measured at the wavelength of 330 nm in the various conditions. The absorption spectrum obtained after long-term storage at room temperature is similar to that obtained after storing the sample for a short time at 100°C. The result suggests that the absorption spectrum of irradiated polycarbonate consists of two components, namely unstable and stable components. By warming the sample up to 100°C the unstable component is almost eliminated, while the stable component still remains.

Temperature dependence of the stability of the optical density during storage is shown in Fig. 10. Obviously the optical density decreases with increasing storage temperature. In this experiment the optical density measurements were made after storing the sample at given temperatures for

one hour. The results of the temperature effects indicate that heat treatment of the irradiated sample is effective for stabilizing the radiation-induced absorption. In order to determine the optimum condition of heat treatment, variation of the optical density with heating period was investigated. As shown in Fig. 11 the optical density is independent of the heating period at 100°C, while it slowly decreases with heating period at 80°C. Consequently, the heating at 100°C for 30 min or more, say 1 h, is the optimum condition for stabilizing the induced absorption.

The effect of irradiation temperature on the response for gamma-rays was also observed as shown in Fig. 12. In this experiment, dose rate was 2 Mrad/h and irradiation time was 3 h. In the shorter wavelength the response becomes seriously sensitive to the temperature and increases with increase of temperature in contrast to the less effect at the longer wavelength. In case of electron irradiation the influence of temperature rise may be negligible, since the irradiation time is very short compared with gamma-ray irradiation. However, temperature rise during irradiation also should be taken into account for high-intensity electron irradiation.

3.4 Calibration of polycarbonate film dosimeter

The absorbed dose calibration of polycarbonate dosimeter for the electron beam was carried out by normalizing the integrated value of a relative depth-dose curve by the energy fluence determined with graphite calorimeter. A polyethylene block with a rectangular depression of 4.5 cm long, 1.3 cm wide, and about 0.7 cm deep was used as a sample holder. A stack 0.7 cm thick of polycarbonate films, each 0.1 mm thick, was set in the depression. An equilibrium condition on electron scattering at the side of polycarbonate film stack is approximately established by this arrangement. The sample was irradiated with beam condition of 1.5 MeV and 1 mA at a distance of 30 cm from the center of beam window. Irradiation time was controlled by beam shutter which operates with high speed. The optical density change at 330 nm in each film was converted into relative absorbed dose, which was read from the relation between optical density change and irradiation time. The energy fluence of incident electron beam was measured by setting the calorimeter at the same position of the polycarbonate stack. The energy fluence was determined from the initial increasing rate of graphite temperature immediately after opening the shutter.

The optical density obtained with each film was covered to relative absorbed dose per unit fluence by means of the following formula:[11)]

$$D_e = \frac{\rho \cdot t \cdot \Sigma J_i}{F} 10^8 \quad , \tag{1}$$

where D_e : relative dose per unit dose ($Mrad^{-1}$)

ρ : density of polycarbonate (1.2 g/cm^3)

t : film thickness (0.1 mm)

J_i : relative absorbed dose in i th film

F : incidence energy fluence (0.75×10^7 erg/cm^2).

By using D_e the absorbed dose in Figs. 13 and 15 are scaled as absolute value as shown in the next paragraph.

The absorbed dose for γ-ray irradiation was determined as follows. The exposure rate was determined by a thin parallel-plate ionization chamber[12] which was designed as a reference dosimeter at high dose-rate and calibrated with the standard ionization chamber at low dose-rate (10^3 - 10^4 R/h). The absorbed dose rate, D_g, in polycarbonate dosimeter was determined by using the relation

$$D_g = \dot{X} \cdot 0.869 \cdot {}_{pc}\mu_{en}/{}_{a}\mu_{en} \ (rad/h), \tag{2}$$

where $\dot{X}$ is the exposure rate in R/h, and ${}_{pc}\mu_{en}$ and ${}_{a}\mu_{en}$ the mass energy absorption coefficients of polycarbonate and air, respectively. The value of ${}_{pc}\mu_{en}$ calculated by Bragg's rule of additivity[14] for each element of polycarbonate ($C_{16}H_{14}O_3$) is 0.0281 cm^2/g for ^{60}Co gamma-rays.

3.5 Absorbed dose measurement

For the determination of absorbed dose by using polycarbonate film the film 0.1 mm thick was selected as a standard sample. Calibration curves for electron dosimetry after storage for one day at room temperature are presented in Fig. 13 and those after heat treatment for 1 h at 100°C are shown in Fig. 14. It is noticed with the 300 nm curve that a linear range is shorter than other curves and there is a large fluctuation for measured opitcal density. The result in Fig. 13 discloses that slopes obtained with gamma-ray irradiation is larger than those with electron irradiation and their differences are about 10 - 20 % irrespective of wavelength. These results suggest that the response is dependent on dose rate as reported by Richold[15]. As can be seen in Fig. 13 the calibration curve in log-log plot for electrons is linear in the ranges of 1 Mrad to 50 Mrad at 330 nm, and 2 Mrad to 100 Mrad at 350 nm. The empirical formula was obtained by least

square method fitted on the linear regions:

$$D_{330} = 4.39 \cdot 10 \cdot (OD)^{1.07} \quad (1 \leqq D \leqq 50) \tag{3}$$

$$D_{350} = 1.41 \cdot 10^2 \cdot (OD)^{1.065} \quad (2 \leqq D \leqq 100) , \tag{4}$$

where OD is optical density increase and D the absorbed dose in Mrad. After heat treatment at 100°C linear regions are extended to 100 Mrad at 330 nm, and to 300 Mrad at 350 nm (see Fig. 14). The dose is given by

$$D_{330} = 5.52 \cdot 10 \cdot (OD)^{1.127} \quad (1 \leqq D \leqq 100) \tag{5}$$

$$D_{350} = 1.52 \cdot 10^2 \cdot (OD)^{1.104} \quad (2 \leqq D \leqq 300) . \tag{6}$$

Practically dose measurements can be made up to 300 Mrad by using the calibration curves measured at 350 nm after heat treatment. When 400 nm is used for dosimetry, measurable upper limit can be extended to 500 Mrad as shown in Fig. 13. However, 500 Mrad irradiation makes the film too fragile for routine use. Each of all calibration points in Figs. 13 and 14 is obtained by means of the readings for three dosimeters irradiated simultaneously. The standard deviation for the range of measuring dose is within 0.1 - 3.5 %. Decrease of the optical density for 6 days after an initial storage period of 24 hr at room temperature is about 2.3 %. The dust and finger prints on the film bring about a little increase in optical density, and this can be eliminated by brushing and washing the samples.

4. Comparison with Other Dosimeters

4.1 Dosimeters for high dose above 10 Mrad

In high dose measurement, almost all of dosimeters which can be practically and reliably used, such as poly(methyl methacrylate), cellulose triacetate film and cobalt glass dosimeters, are mainly intended for the measurement at relatively lower dose region (<10 Mrad). Though many dosimetric methods for high dose region (>10 Mrad) have been reported, most of them are not practical, or the detailed properties have not been clarified.

Main methods used for the measurement of high dose above 10 Mrad are listed in Table 1. Aqueous chemical dosimeters are not generally useful because of the convenience. The semiconductor dosimeter and the glass dosimeters listed are free from oxygen effect which usually causes disadvantages in plastic film dosimeters, but their reliabilities are unknown

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and they are not commercially available. Hydrogen pressure dosimeter may be most reliable methods in Mrad to Grad dosimetry at present; it is independent of dose rate and free from some defects inherent in solid-state and chemical dosimeters. For the routine measurement, however, it is not so convenient compared with plastic film dosimeters using optical density measurement.

Plastic film dosimeters are considered to be the most practical of all dosimeters even for high level dosimetry (>10 Mrad) as long as the influence of fading, temperature, oxygen and other chemicals are small.

4.2 Comparison with poly(ethylene terephthalate)(PET) film dosimeter and cellulose triacetate (CTA) film dosimeter

As shown in Table 1, there are several types of plastic film dosimeters for the measurement of high dose above 10 Mrad. We compared the properties of polycarbonate film dosimeter with those of other plastic film dosimeters, PET and CTA which have been already used in several irradiation facilities. The thickness of the PET film and the polycarbonate film used for this comparison is about 0.05 and 0.1 mm, respectively.

General tendencies of fading after short time irradiation at room temperature in air are shown in Fig. 15. The fading rate of polycarbonate film dosimeter is very slow compared with PET film dosimeter and the absolute quantity of the optical density change is almost the same as that of CTA film dosimeter.

The relation between optical density change of PET film dosimeter and the absorbed dose for electron and gamma radiation is shown in Fig. 16, where the optical density was measured after the post-irradiation storage of about one day. It is a linear function of dose in higher dose region above 100 Mrad. The maximum measurable dose is about 1000 Mrad for electron irradiation, though the irradiation above 500 Mrad makes the film fragile. In the case of CTA film, the optical density change at 280 nm increases linearly with dose until about 20 Mrad and the upper limit of the measurable dose is about 30 Mrad. The measurement of still higher dose may be possible by using large wavelength (300 nm). Practically, however, CTA dosimeter is not suitable for the higher dose measurement than 30 Mrad because of radiation induced fragility. Thus, PET dosimeter is the most resistant for radiation.

A comparison of three films on dosimeter responses for gamma rays (1 Mrad/h) and electron beam (1000 Mrad/h) is shown in Fig. 17. The measurement wavelength of polycarbonate and PET film dosimeter is 330 nm. The

difference in sensitivity between gamma rays and electron beam is ascribed to dose rate dependence. It is the smallest in polycarbonate film dosimeter, and the largest in PET film dosimeter. In the latter, the sensitivity for 1 Mrad/h is about twice as much as that for 1000 Mrad/h.

Table 2 lists some properties of the three dosimeters: the thickness which is suitable for usual dosimetry, the suggested wavelength for spectrophotometry, the approximate measurable dose range, the approximate ratio of the sensitivity at 1 Mrad/h to that at 1000 Mrad/h (dose rate dependence), the linearity in dosimeter response, the fading for a day storage (in the case of initial optical density change = 0.3, electron irradiation), and the irradiation temperature dependence for gamma-ray around room temperature. It is found from this table that polycarbonate film dosimeter is the most useful for the dose range from 10 Mrad to 300 Mrad.

5. Summary

A polycarbonate film 0.1 mm thick was found to be useful dosimeter as a simple method of high-level dosimetry. Optical density change in the wavelength of 330 nm and 350 nm were chosen for dosimetry by considering dose range and fading. The measurable dose range is 1 - 100 Mrad at 330 nm and 2 - 300 Mrad at 350 nm. The standard deviation derived from three samples is less than 3.5 %.

The radiation-induced optical density rapidly decreases during the first 1 day after irradiation and thereafter the subsequent fading is small amounting to about 0.4 % per day at 330 nm at room temperature. To reduce the error in measurements of optical density a storage time must be allowed at least 1 day after irradiation.

The decrease of optical density is sensitive to storage temperature. The fading rate is larger at higher temperature than lower temperature and also it depends on the wavelength and is the smallest at 330 nm. Therefore, if all the measurements are made at 330 nm, very little fading will be observed even up to several days after irradiation. Since fading is a complex function of dose, film thickness, initial optical density, and measurement wavelength, it is very difficult to give a simple formula on the decrease of the optical density. Optical density can be stabilized by a post-exposure heat treatment. For practical dosimetry a heat-treatment for 1 h at 100°C was found to be the most convenient.

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The radiation-induced optical density rapidly decreases during the first 1 day after irradiation and thereafter the subsequent fading is small amounting to about 0.4 % per day at 330 nm at room temperature. To reduce the error in measurements of optical density a storage time must be allowed at least 1 day after irradiation.

The decrease of optical density is sensitive to storage temperature. The fading rate is larger at higher temperature than lower temperature and also it depends on the wavelength and is the smallest at 330 nm. Therefore, if all the measurements are made at 330 nm, very little fading will be observed even up to several days after irradiation. Since fading is a complex function of dose, film thickness, initial optical density, and measurement wavelength, it is very difficult to give a simple formula on the decrease of the optical density. Optical density can be stabilized by a post-exposure heat treatment. For practical dosimetry a heat-treatment for 1 h at 100°C was found to be the most convenient.

The effect of irradiation temperature was negligible around room temperature, though it has not been proved at various irradiation conditions. Oxygen effect on optical density change also seems to be negligible with preliminary experiment. In addition to this problem, the coloration mechanism in near ultra-violet region must be investigated by future study.

Polycarbonate film dosimeter was compared with other similar film dosimeters in some dosimetric properties, and it has proved to be more useful for high dose range from 10 Mrad to 300 Mrad than PET film dosimeter and CTA film dosimeter.

Acknowledgements

The authors wish to thank the members of Irradiation Service Section for their kind help in irradiating the samples.

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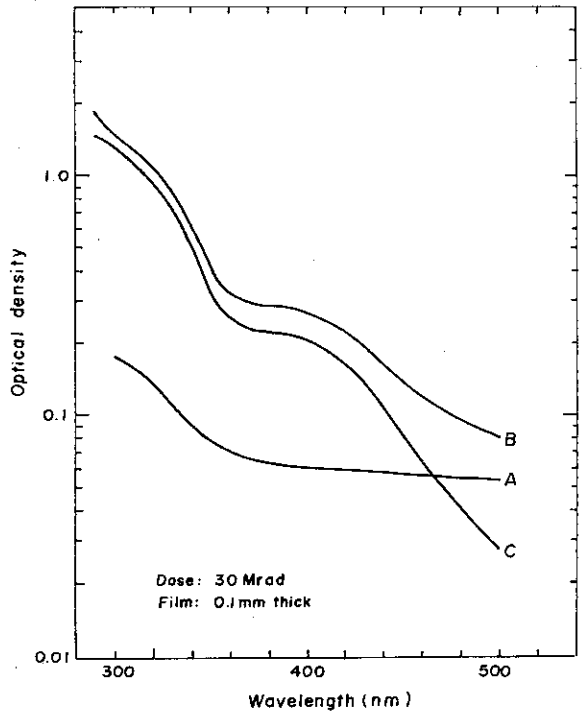

Fig. 1 Typical absorption spectra of the polycarbonate film, A, unirradiated; B, irradiated; C, difference between A and B.

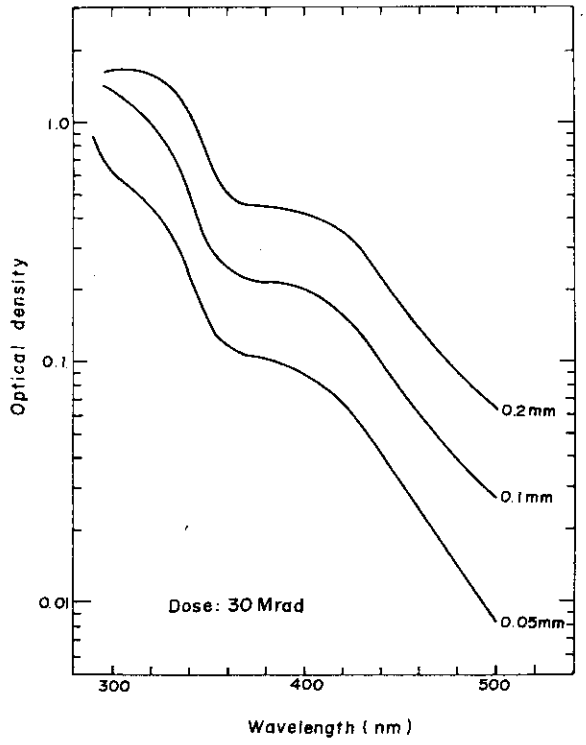

Fig. 2 Absorption spectra of the polycarbonate films of different film thickness irradiated to 30 Mrad.

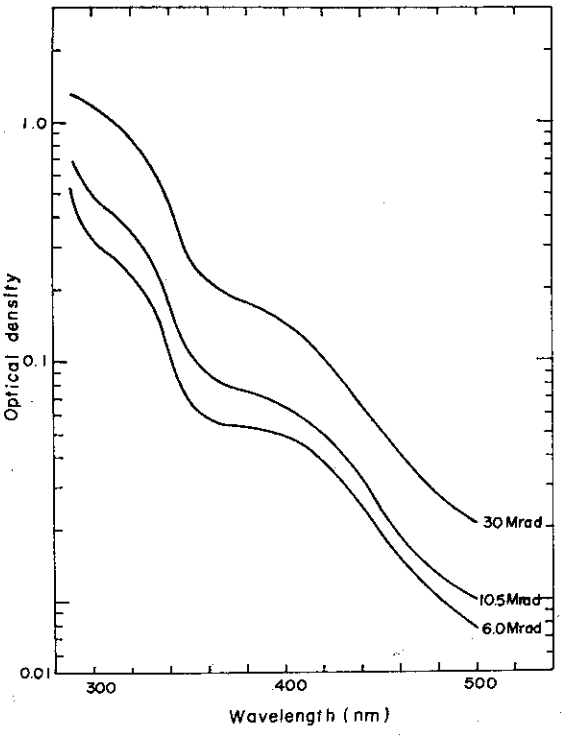

Fig. 3 Absorption spectra of the polycarbonate films of 0.1mm thick irradiated to different dosage.

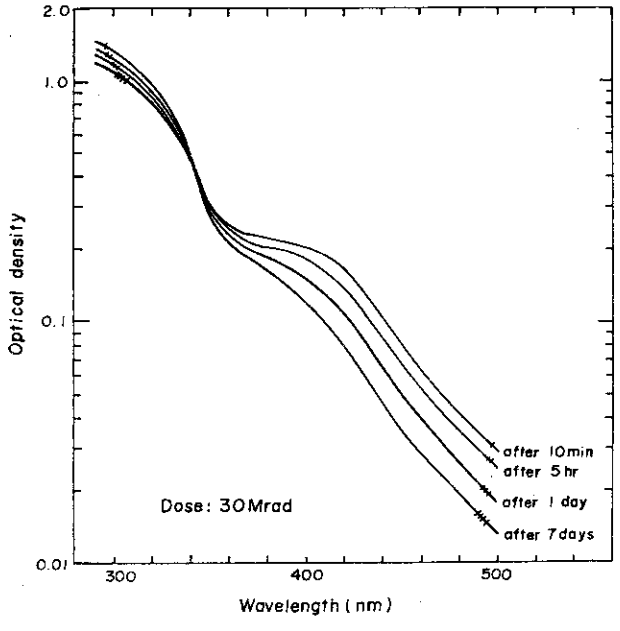

Fig. 4 Radiation induced spectral change for 0.1mm thick film kept at room temperature.

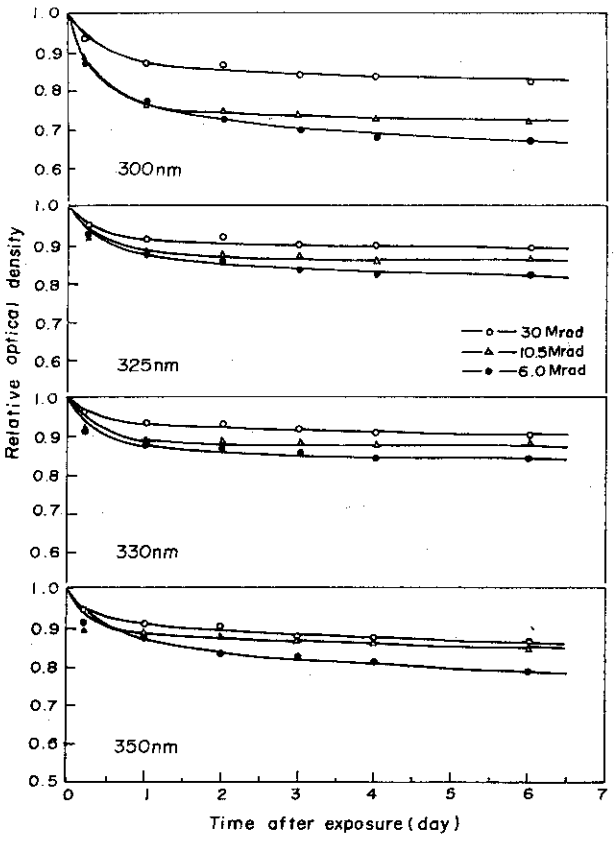

Fig. 5 Post-irradiation fading at room temperature for given wavelength.

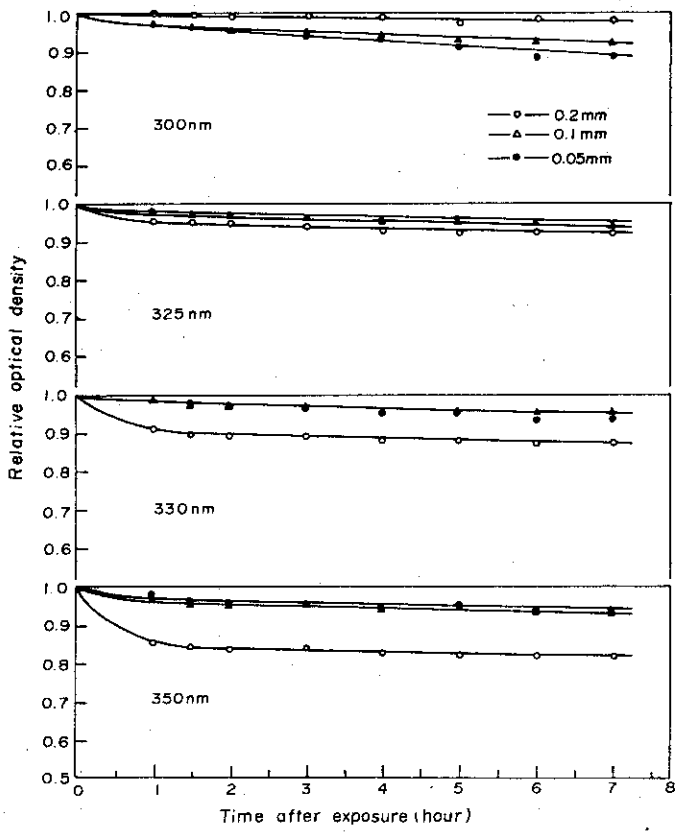

Fig. 6 Post-irradiation fading at room temperature.

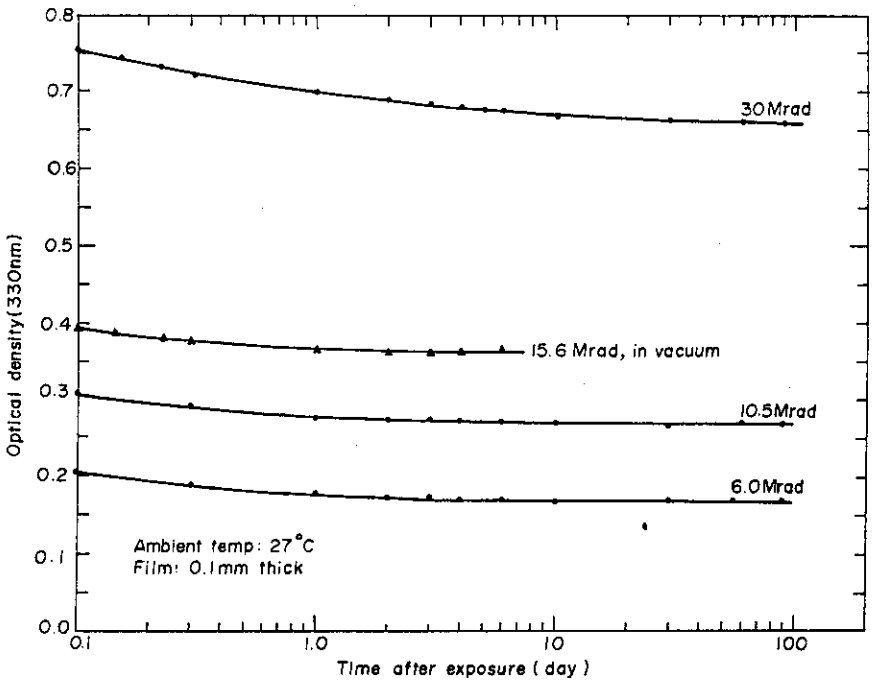

Fig. 7 Fading of optical density for various doses at room temperature.

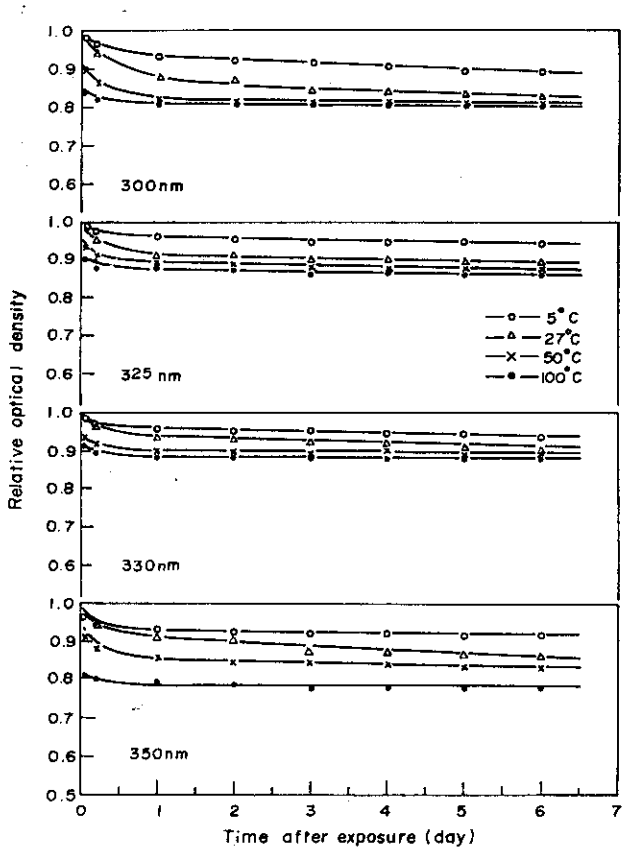

Fig. 8 Fading of optical density for various ambient temperatures. (Dose; 30 Mrad, film; 0.1mm thick)

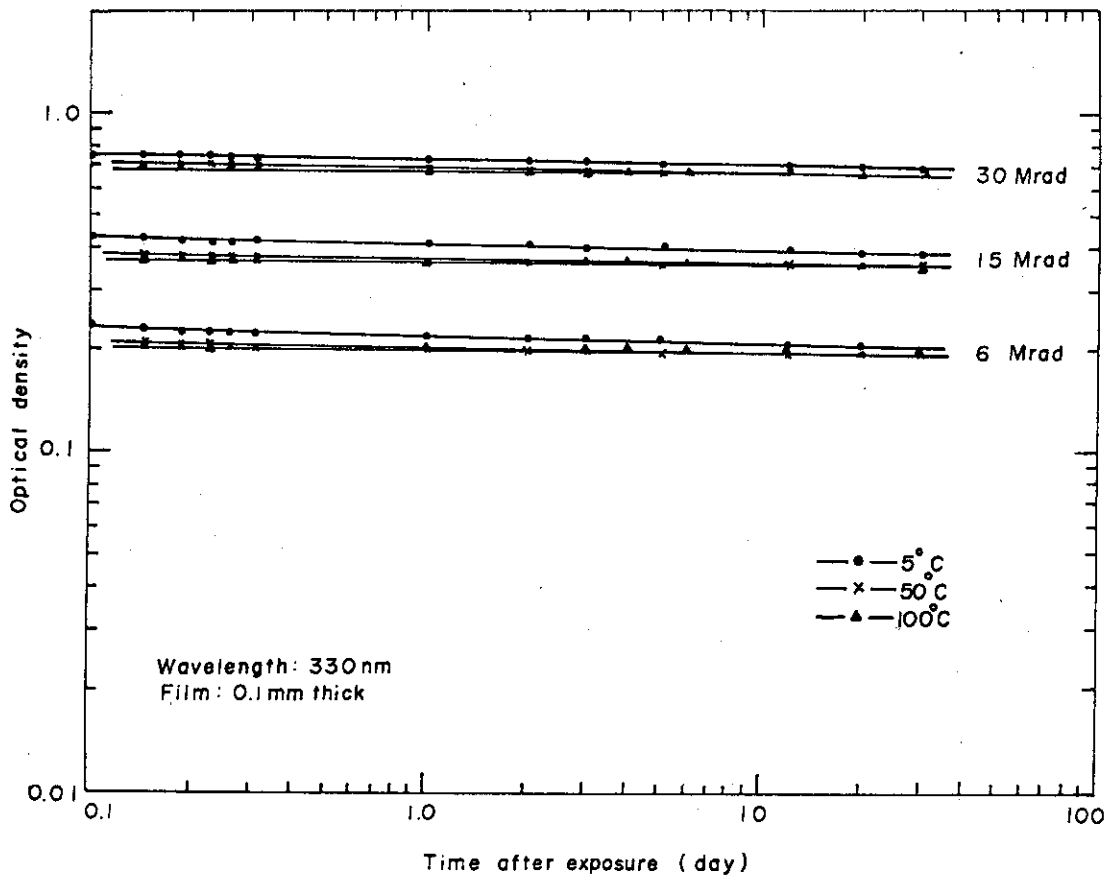

Fig. 9 Fading of optical density for various doses at given ambient temperatures.

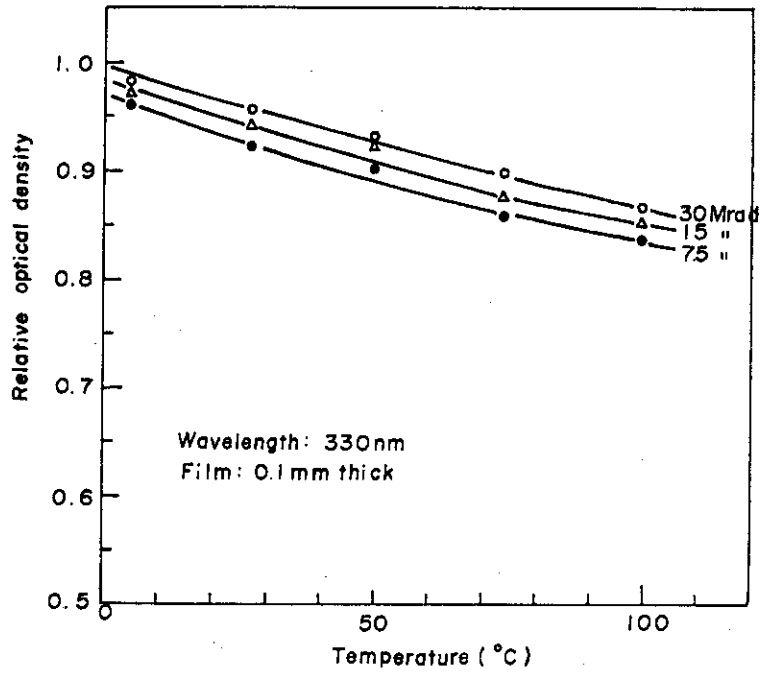

Fig. 10 Changes in optical density after storage of 1 h with temperature.

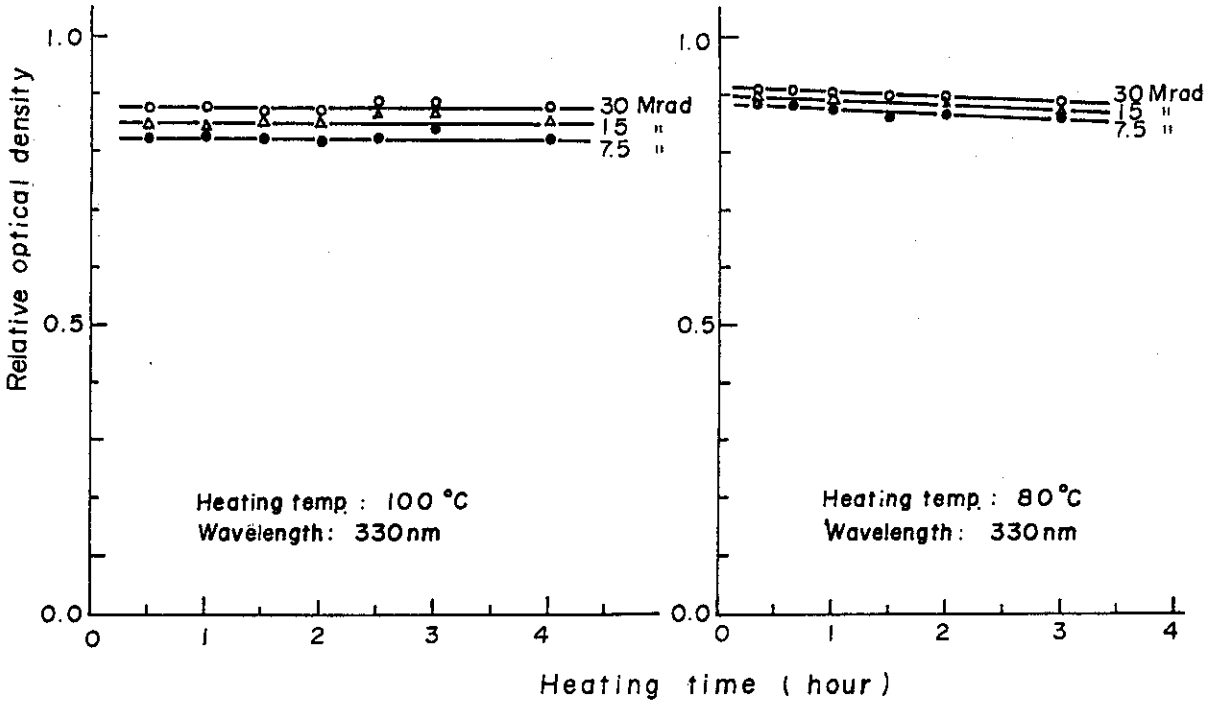

Fig. 11 Influence of heat treatment period on the optical density at 330nm.

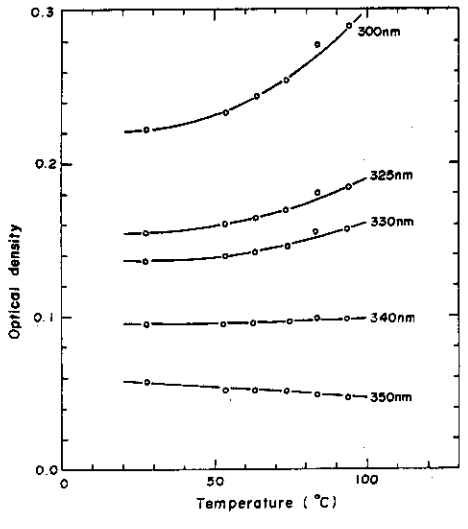

Fig. 12 Effects of irradiation temperature on optical density response (gamma-rays)

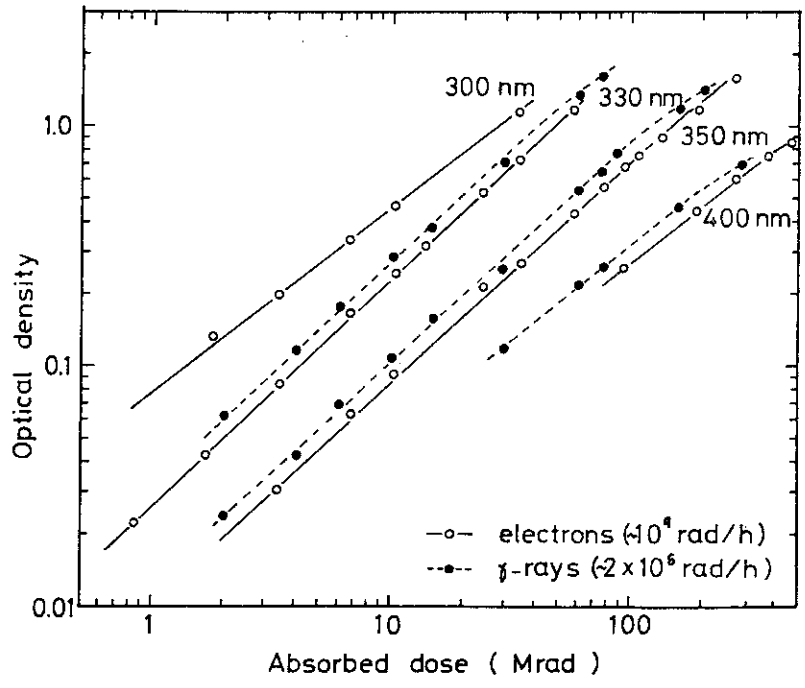

Fig. 13 Dose calibration curves for 0.1 mm thick film for the storage of one day at room temperature.

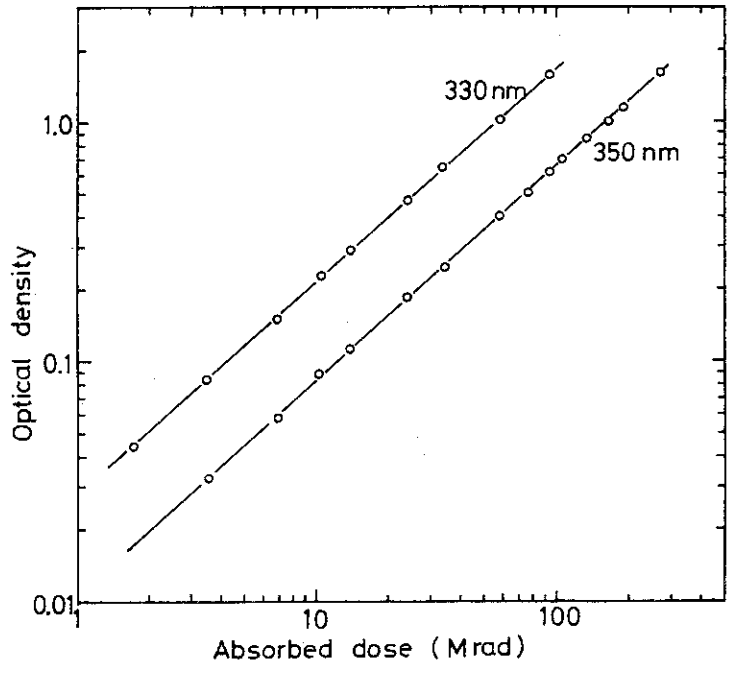

Fig. 14 Dose calibration curves for 0.1 mm thick film after heat treatment at 100°C for 1 h.

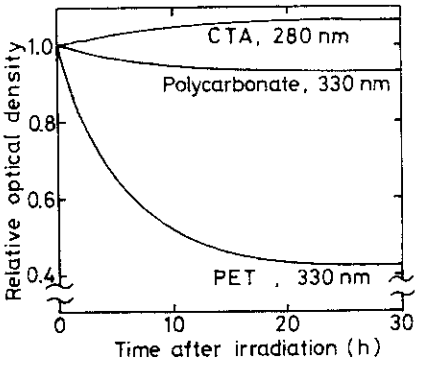

Fig. 15 General tendencies of fading of three plastic film dosimeters after short time irradiation at room temperature in air.

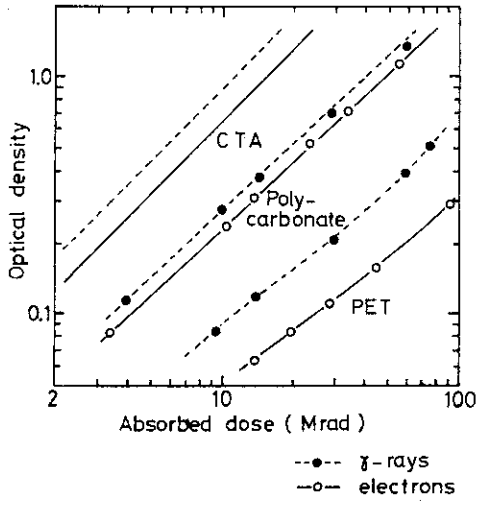

Fig. 17 A comparison between three plastic film dosimeters on dosimeter response for gamma rays (1 Mrad/h) and electron beam (1000 Mrad/h).

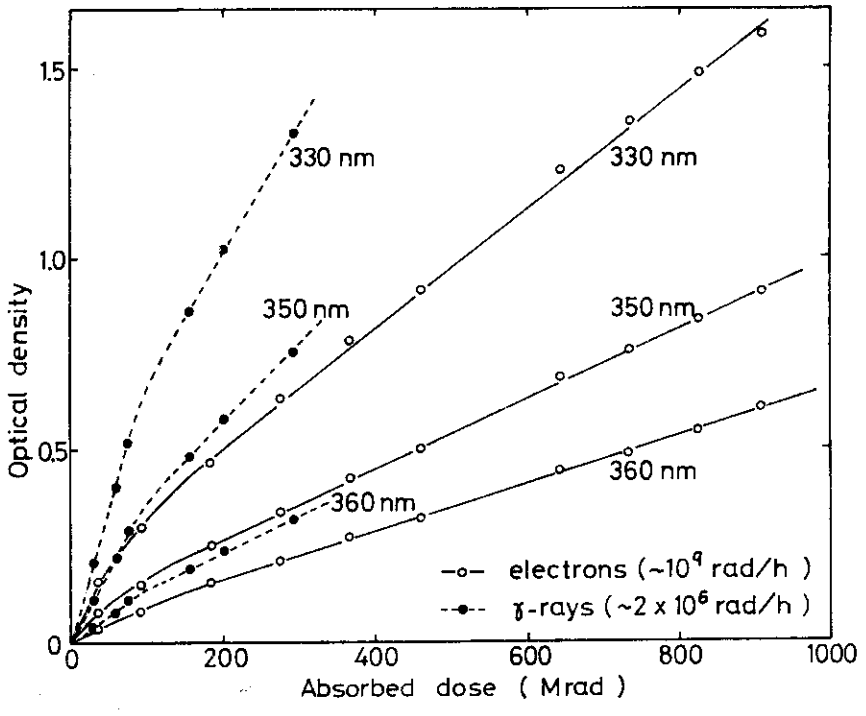

Fig. 16 The relation between optical density change of PET film dosimeter and the absorbed dose for electron and gamma radiations.