Chemical Etching Studies of a Brazilian Polycarbonate to Fast Neutron Detection

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Abstract

The Dosimetric Materials Laboratory (LMD) of the Radiation Metrology Center (CMR) is developing a personal dosimeter for fast neutrons using the technique of solid state nuclear track detectors (SSNTD). This technique is based on the recorded damage (tracks) in dielectric materials due to the impact of charged particles. The tracks are revealed and amplified for visualization in optic microscope through a technique known as chemical etching. The LMD is investigating a Brazilian commercial polycarbonate as a new passive fast neutron's detector in substitution to the traditional materials, as the cellulose nitrate LR-115 and the polycarbonates Makrofol and CR-39. The variation of the etching parameters (chemical solution, time and temperature) alters the response of the material; the best revelation conditions provide the best relationship among the amount of revealed tracks, their clearness and the time spent for this. The polycarbonate studied is a resin of same chemical monomer of Makrofol ($C_{16}H_{14}O_3$). Samples of 3 x 1 cm^2 of the polycarbonate were irradiated with 5 mSv of fast neutrons (^{241}Am-Be) and revealed with the chemical solution PEW-40 (15% KOH, 45% H_2O, 40% C_2H_5OH), commonly used for Makrofol. The studied etching parameters were time and temperature. Groups of four samples were revealed at temperatures of 50, 65, 75, 90 and 100ºC with etching times varying from one to six hours. The used track's counting procedure was that referred in the literature. The best response to fast neutrons was obtained at 75ºC; in spite of their similar answers, smaller temperatures join larger uncertainties in the track's counting and poorer clearness. At this temperature, the number of revealed tracks increases with the etching time approximately until a plateau at three hours. For etching times higher than four hours the polycarbonate presents overlap of tracks. If the temperature is adjusted to 75ºC, the etching time should be in the plateau to avoid that small time errors alter significantly the counting of tracks. On the other hand, besides the tracks overlapping that implies in tracks counting errors, sensitivity curve plateau is reached for a higher elapsed time compared to the working day of a dosimetry laboratory.

1. INTRODUCTION

The Dosimetric Materials Laboratory (LMD) of the Radiation Metrology Center (CMR) is developing a personal dosimeter for fast neutrons using the technique of solid state nuclear track detectors (SSNTD). This technique is based on the recorded damages (tracks) in dielectric materials due to the impact of charged particles [1,2,3]. When neutrons with energies higher than 1 MeV collide with carbon and oxygen atoms with compose the polycarbonate molecule, recoil nuclei are liberated, which will produce the tracks. The tracks are revealed and amplified for visualization in optic microscope through a technique known as chemical etching [4]. In this procedure, fine hollow channels are formed along the recoil nuclei paths.

In a previous study a Brazilian commercial polycarbonate was presented as a potential new passive fast neutron's detector in substitution to traditional materials [5], as the cellulose nitrate LR-115 and the polycarbonates Makrofol and CR-39. Considering that this is a new material, it is necessary to characterize its performance before starting its use.

The motivation for this study was to obtain the best response of the new track detector for neutrons. The best response is correlated with the best revelation and amplification conditions of neutron tracks. This is determined by the study of the variation of the chemical etching parameters: chemical solution, etching time and etching temperature. It provides the best relationship among the amount of revealed tracks, their clearness and the time spent for this.

2. EXPERIMENTAL PROCEDURE

2.1. Polycarbonate description

The material studied is a Brazilian commercial polycarbonate manufactured for use in civil construction and industry and for military use (policy shields) [5]. However it has the same chemical monomer of Makrofol ($C_{16}H_{14}O_3$), well-known as passive track detector for fast neutrons [6,7].

Each detector corresponds to a sample of 3 x 1 cm^2 of a 1.5 mm thick sheet of this resin. A mark is made in the upper left corner of each sample for face identification.

2.2. Irradiation procedure

The samples were irradiated using an isotropic ^{241}AmBe (3,7 GBq) source of the Nuclear Engineering Center (CEN) of IPEN, in free air and ambient temperature. The experimental arrangement consists of a suspended circular metallic bracket 1500 mm far from the floor, with the source located in the center. The detectors were placed on a ring 20 cm distant from the source, always with the sensitive face at normal incidence. A hundred and sixteen detectors were irradiated with dose of 5 mSv.

2.3. Chemical etching

This procedure consists of immersing the polycarbonate detectors, individually, in test tubes with a chemical solution inside a laboratory electric oven at given temperature during an established time. The chemical solution used was the PEW-40 (15% KOH, 45% H_2O, 40% C_2H_5OH), commonly used for Makrofol [8]. The etching was stopped by rinsing the detectors into running water.

As the chemical solution PEW-40 is well-known for this application, the studied etching parameters were time and temperature. The chemical etching found in the literature uses 70ºC; however there is no good temperature stabilization of IPEN laboratory electric oven at temperatures bellow 75ºC, then the etchings at temperatures below this were carried out at the laboratory of PRO-RAD Ltda. Groups of four samples were revealed at temperatures of 50, 65, 75, 90 and 100ºC with etching times varying from one to six hours, with increments of one hour. For the best etching temperature, different times have been studied for better knowing the etching time behavior.

2.4. Track counting

The used track's counting procedure was that referred in the literature [9]. The detectors surfaces were visualized through a video camera Samsung SDC-312ND connected in an optical microscope Optovac OptoNTIM. The microscope magnification used was 160:1.

The counting area is defined by fields of 20 x 0.1 mm^2 (band) of the detector surface. The camera vision field defines this 0.1 mm width. The track density of a polycarbonate is the arithmetic mean of five different bands counting and the uncertainty is the mean standard deviation.

3. RESULTS AND DISCUSSION

The track density for each time and temperature studied is presented in Table 1. With etching time of one hour the counting is similar to background detector counting (detectors not irradiated), except for 90 and 100ºC. These tracks are very small, but they stand out due to its strong coloration. The counting for the largest temperature demonstrates a very poor revelation condition, probably due to the strong evaporation of the chemical solution, which for 5 and 6 hours etching was sufficient to emerge part of the detectors during the procedure.

Table I. Neutron Track Density for different chemical etchings

Time (h)	*50ºC (tracks/cm²)*		*65ºC (tracks/cm²)*		*75ºC (tracks/cm²)*		*90ºC (tracks/cm²)*		*100ºC (tracks/cm²)*	
	BG		*BG*		*BG*		*BG*		*BG*	
1	0 ± 0	0 ± 0	0 ± 0	40 ± 52	10 ± 21	15 ± 24	67 ± 36	395 ± 64	15 ± 23	118 ± 56
2	0 ± 0	490 ± 139	0 ± 0	1830 ± 410	35 ± 34	1385 ± 221	43 ± 26	585 ± 259	54 ± 24	204 ± 64
2.5	-	-	-	-	25 ± 26	2275 ± 319	-	-	-	-
3	19 ± 25	1460 ± 601	19 ± 25	2145 ± 645	60 ± 46	2600 ± 386	144 ± 38	840 ± 320	57 ± 64	219 ± 53
4	36 ± 45	2205 ± 525	74 ± 22	2260 ± 1057	55 ± 44	2515 ± 397	178 ± 51	1535 ± 256	33 ± 33	143 ± 78
5	48 ± 43	2055 ± 524	80 ± 28	2015 ± 964	0 ± 0	2470 ± 422	53 ± 43	1735 ± 217	-	-
6	54 ± 49	1840 ± 278	70 ± 57	2020 ± 664	0 ± 0	2405 ± 409	58 ± 27	1630 ± 203	-	-

After 4 h etching, the tracks showed low contrast with the surface not impinged by neutrons, being visually weak (Figure 1a and 1b) and some overlap of tracks (Figure 1c) start to be seen. The overlap phenomenon appears to increases with time.

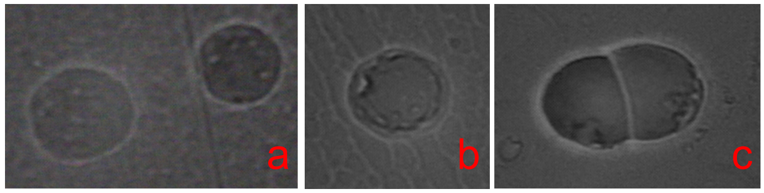

Figure 1. Neutron tracks in the polycarbonate: a-b) low contrast track; c) track's overlap

The curves of Figure 2 show the behavior of polycarbonate sensibility for the different chemical etching conditions. One could note similar shapes: a slope in the beginning and a plateau. The etching time should be adjusted in the plateau region or close to it to avoid small time errors from altering significantly the counting of tracks, even if there is a good sensitivity and clearness of tracks on the slope portion of the curve. For this reason temperatures of 50 and 90ºC cannot be used, because the plateau is reached after four hours. This is far much time if added with the necessary time to stabilize the laboratory electric oven; implicating on an elapsed time higher than the working day of a dosimetry laboratory.

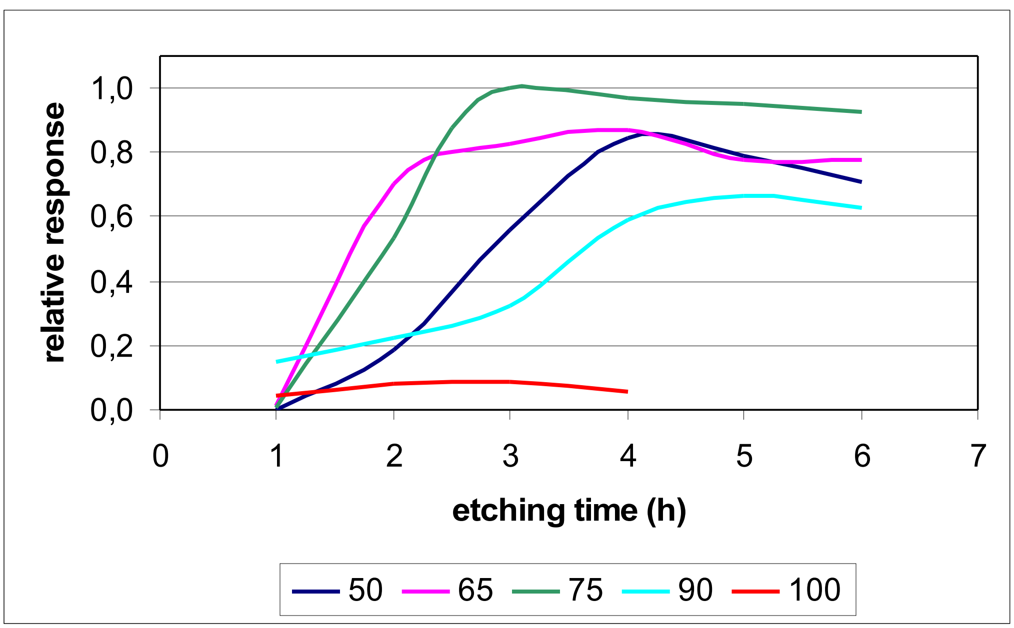

Figure 2. Polycarbonate sensibility to different etching times and temperatures

The best response was obtained at 75ºC, not only for the higher track counting on the plateau, but also because the smaller uncertainties associated to countings (Table I). By adjusting the etching temperature to 75ºC, the plateau is reached approximately in 3 h. It is well true that at 2.5 h there is a very good relationship among the amount of revealed tracks and their clearness (Figure 3). Before 4 h the polycarbonate presents the problems described above.

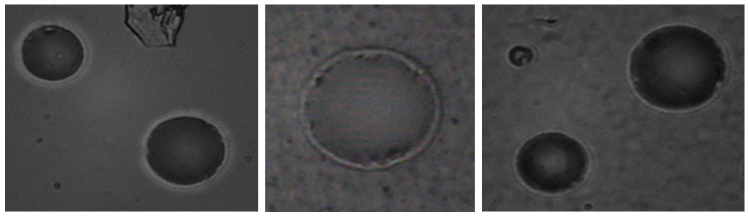

Figure 3. Typical Neutrons tracks at 75oC, 2.5 h

4. CONCLUSIONS

Among the studied etching temperatures, 75ºC showed the best response for ^{241}Am-Be neutrons. Other temperatures join larger uncertainties in the track's counting or the time spent to reach the plateau of the curve is high for a working day of a dosimetry laboratory. At this temperature, the counting is more accurate and the number of revealed tracks is greater than at other temperatures. From 2.5 h to 4 h one can obtain the best chemical etching condition due to the good track's clearness presented.

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