THINK INDIA JOURNAL

ISSN: 0971-1260

Vol-22-Issue-17-September-2019

Thermal annealing effects on bulk etching characteristics in polycarbonate detectors

Jeeban Pd Gewali[1*] and Bimla Jaishy[2]

[1]Department of Physics, Lovely professional University, Punjab

[2]Department of Physics, Assam Don Bosco University, Guwahati

*Email: jeeban.21059@lpu.co.in

Abstract

Solid state nuclear track detectors (SSNTD) are essentially dielectric materials which are important in the study of interactions of heavy ions with matter. The properties of the dielectric material play an important role in determining the nuclear track parameters. In this context we study the variation in bulk etching property of Makrofol-E and Lexan polycarbonate detectors and its variation when annealed at temperatures 80^{o}C, 100^{o}C, 120^{o}C, 140^{o}C and 150^{o}C.

Key Words: Solid State Nuclear Track Detectors, Makrofol-E, Lexan, Chemical Etching, Annealing, Bulk-etch rate, Activation energy

1. Introduction

A study of etching characteristics of a SSNTDs which is the property of a detector itself, one may be able to optimize the usefulness of track detectors [1,2]. In this work we have used Makrofol-E and Lexan polycarbonate detectors for studying their bulk etching characteristics. It is well established that thermal annealing reduces track etch rate (V_T) and bulk etch rate (V_G) of the ion in the detector by partial repairing of damage trail. Here we study the effect of thermal annealing on the detector itself. Thermal annealing of two polycarbonate detectors Makrofol-E and Lexan has been studied by measuring thickness of the removed layer during etching of the detector. [3, 4]

ISSN: 0971-1260

Vol-22-Issue-17-September-2019

For detector bulk etch rate V_G perpendicular to the surface is given by

$$V_G = \left({}^{(X_i - X_f)}/_{2t} \right) \quad (1)$$

where X_i and X_f are initial and final measured thickness of the detector etched for time t in an etchant at constant temperature.

2. Experimental Procedure [3-6]

Table below gives the properties of two polycarbonate detectors Makrofol-E and Lexan used in the study.

Detector	Chemical Name	Chemical Formula	Thickness (μm)	Density (g cm^{-3})	Colour
Makrofol-E	Polycarbonate	$C_{16}H_{14}O_3$	405	1.14	Colourless
Lexan	Polycarbonate	$C_{16}H_{14}O_3$	315	1.20	Colourless

2.1. Thermal Annealing and Chemical etching of the detectors

Small rectangular pieces of Makrofol-E and Lexan detectors were cut and annealed at 80°, 100°, 120°, 140° and 150°C respectively for 10 min in a heating oven maintained at constant temperature having an accuracy $\pm$ 1° C.

The chemical etching was carried out in 6.0N NaOH at 55°C in a constant-temperature water bath, having an accuracy $\pm$ 0.5° C.

2.2. Thickness Measurement

Thicknesses of the detector were measured in a Motic optical microscope at 10X object magnification placing it in a sample holder. Error mentioned in the table for surface removed are the standard deviation error which is ranging between $\pm$ 0.4 to 1.4 μm.

ISSN: 0971-1260

Vol-22-Issue-17-September-2019

2.3. Measurement of Bulk etch rate

The rate at which the undamaged surface of the detector is removed during etching process is called the bulk etch rate V_G. Due to the chemical reaction between etchant and the detector material, some molecules of the detectors are removed which leads to the removal of the material from the detector surface. Thus a detector during etching process becomes smaller and smaller as the material is removed layer by layer. [4]

In this work to study the etching effect, two polycarbonate detectors Makrofol-E and Lexan are (i) Etched at different temperature and (ii) annealed between 80 to 150^{o}C are chemically etched with 6N NaOH at 55^{o}C. The concentration of NaOH was kept at 6N.

After etching the detectors thoroughly washed with dematerialized water and after drying it in desiccator its thickness was measured in optical microscope. These operations were repeated many times to find the change in thickness at different etching time and V_G was obtained in units of length per unit time as

$$V_G = {\Delta X}/{2t} \qquad (2)$$

where ΔX is the change in thickness due to etching time for a time period t.

3. Activation Energy

The activation energy is defined as the energy barrier that the reactants must overcome in order to react. It can also be defined as the minimum energy needed to activate the molecules to undergo a phase transition. The variation of V_G with annealing temperature was found to be exponential and follows the Arrhenius equation [4, 6].

$$\mathbf{V_G = A\ exp\ (-\frac{E_a}{kT})} \qquad (3)$$

where, A being a constant, k Boltzmann's constant, E_a the activation energy and T the annealing temperature.

4. Result and Discussion

ISSN: 0971-1260

Vol-22-Issue-17-September-2019

Table 1 list the total surface etched out for Makrofol-E and Lexan detectors when etched in 6N NaOH at different etching temperature 40, 55, 70 and 80^{o}C. The tabulation shows the maximum surface is etched out when the etching temperature is higher during the same time period, which implies that bulk etching is directly proportional to etching temperature. The surface removed after etching 10 hours for Makrofol-E detector at 70^{o}C is ~3.5 times that at 40^{o}C, similarly for Lexan detector for the same time period surface removed when etched at 80^{o}C is > 5 times when etched at temperature 55^{o}C.

It's a known fact that track length and track diameter and its corresponding track etch rate and bulk etch rate of the irradiated ion decreases with annealing for any detector. [4,5] In this paper we studied the effect of annealing on the bulk etch rate which is also a function of thickness removal with etching time. The experimental data suggests V_G value dips with increase in annealing temperature. It can be observed from the table 2, 3 Initial etching for 4-6 hours does not show any considerable change in thickness. With the increase in etching time the difference on thickness removal is seen. Thickness removed after etching when sample is annealed at 80, 100 and 120^{o}C is not so predominant, the effect of annealing is clearly visible above 120^{o}C where the V_G value drops considerably as reported in table 4. Table 4 rightly depicts the decrease in bulk etch rate with the increase in annealing temperature. It can be seen in case of Makrofol-E V_G at annealing temperature T=150^{o}C is ¼th of the unannelaed detector. Similarly for the case of lexan V_G at unannealed is ~3 times that of V_G annealed at 150^{o}C. Table 4 also lists the value of activation energy for both Makrofol-E and Lexan detectors, which are obtain from the logarithmic variation of bulk etch rate with inverse of annealing temperature as plotted in figure 1. The slope of curve obtained gives the activation energy for surface bulk etching ~ 9.2 Kj mol^{-1} for both the detectors as reported in table 4.

Table 1: Total Surface etched out for Makrofol-E and Lexan detectors, etched in 6N NaOH.

Etching time (hr)	Total Surface Removed (μm)					
	Makrofol-E			Lexan		
	Etching Temperature (^{o}C)			Etching Temperature (^{o}C)		
	40^{o}C	55^{o}C	70^{o}C	55^{o}C	70^{o}C	80^{o}C
1.0	1.3±0.4	1.62±0.4	2.0±0.4	2.2±0.4	5.0±0.4	14.9±0.4

ISSN: 0971-1260

Vol-22-Issue-17-September-2019

2.0	2.6±0.4	3.0±0.4	9.0±0.4	3.6±0.4	9.8±0.4	24.6±0.4
3.0	3.6±0.4	4.2±0.4	12.0±0.4	5.3±0.4	12.9±0.4	31.9±0.4
4.0	4.6±0.4	5.8±0.4	16.0±0.4	6.7±0.4	15.7±0.4	36.7±0.4
5.0	6.0±0.9	6.7±1.0	18.7±1.0	8.0±0.9	17.9±1.4	39.9±1.4
6.0	7.2±0.9	8.0±0.9	22.5±2.0	9.0±0.9	21.2±1.3	46.3±1.4
7.0	8.6±0.9	9.4±0.9	28.0±1.1	9.9±0.9	23.9±1.3	52.6±1.1
8.0	9.8±0.9	10.4±0.9	30.0±1.4	10.8±0.9	26.2±1.2	56.2±1.3
9.0	10.9±0.9	11.8±0.9	32.0±1.0	11.6±0.9	28.7±1.1	59.6±1.4
10.0	11.8±0.9	13.0±0.9	37.5±1.0	12.6±0.9	31.0±0.9	64.5±1.4

*the error indicated is standard deviation

Table 2: Total Surface etched out for Makrofol-E dtectors, etched in 6N NaOH at 55^{o}C**.**

Etching Time (hr)	Total Surface Removed (μm)					
	Annealing Temperature					Unannealed
	150^{o}C	140^{o}C	120^{o}C	100^{o}C	80^{o}C	
1.0	0.2±0.4	0.4±0.4	0.4±0.4	1.1±0.4	1.2±0.4	1.62±0.4
2.0	0.7±0.4	0.9±0.4	1.2±0.4	2.6±0.4	2.9±0.4	3.0±0.4
3.0	1.1±0.4	1.5±0.4	1.9±0.4	2.4±0.4	4.1±0.4	4.2±0.4
4.0	1.9±0.4	2.2±0.4	2.7±0.4	3.7±0.4	5.6±0.4	5.8±0.4
5.0	3.2±0.4	3.6±0.4	4.0±0.4	5.2±0.4	6.3±0.4	6.7±1.0
6.0	3.4±0.9	3.9±0.9	4.4±0.9	5.6±0.9	7.8±0.5	8.0±0.9
7.0	3.8±0.9	4.2±0.9	4.8±0.9	5.9±0.9	8.7±0.9	9.4±0.9
8.0	4.8±0.9	5.1±1.1	5.6±1.1	6.7±1.2	9.8±1.1	10.4±0.9
9.0	5.9±0.9	6.2±1.1	6.6±1.1	7.4±1.2	10.3±1.1	11.8±0.9
10.0	6.7±1.1	7.0±1.1	7.6±1.1	8.5±1.2	11.7±1.2	13.0±1.1

*the error indicated is standard deviation

Table 3: Total Surface etched out for Lexan detectors, etched in 6N NaOH at 55^{o}C**.**

Etching Time (hr)	Total Surface Removed (μm)					
	Annealing Temperature					Unannealed
	150^{o}C	140^{o}C	120^{o}C	100^{o}C	80^{o}C	
1.0	0.4±0.4	0.6±0.4	0.8±0.4	1.9±0.4	2.0±0.4	2.2±0.4
2.0	0.8±0.4	1.1±0.4	1.6±0.4	2.7±0.4	3.3±0.4	3.6±0.4

ISSN: 0971-1260

Vol-22-Issue-17-September-2019

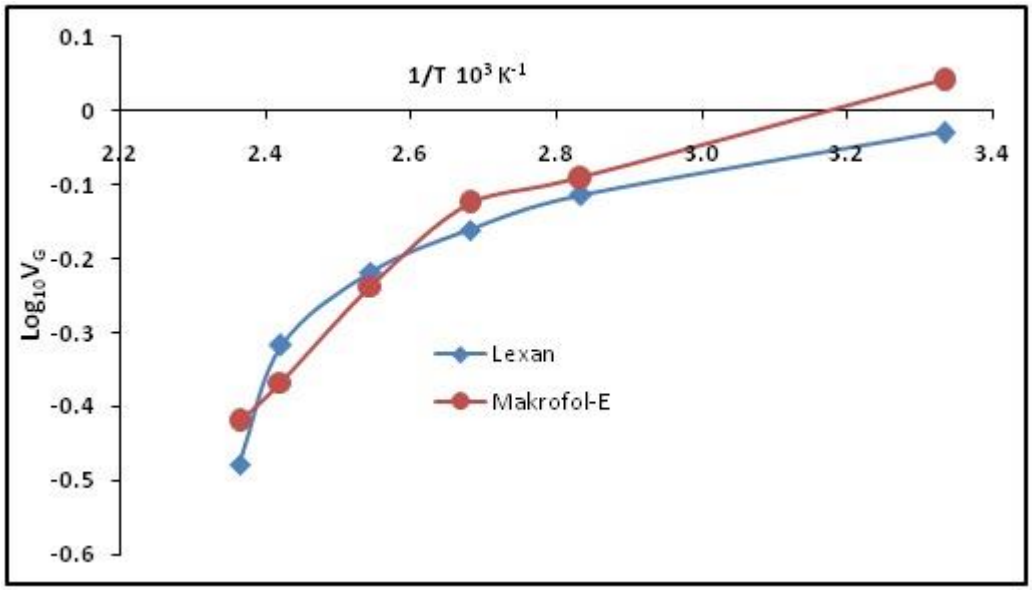
3.0	1.6±0.4	1.9±0.4	2.4±0.4	3.9±0.4	4.7±0.4	5.3±0.4
4.0	2.4±0.4	2.8±0.4	3.2±0.4	4.7±0.4	6.1±0.4	6.7±0.4
5.0	3.1±0.4	3.6±0.4	4.0±0.4	5.9±0.4	7.4±0.4	8.0±0.4
6.0	3.6±0.9	4.1±0.9	4.6±0.9	6.8±0.9	8.6±0.9	9.0±0.9
7.0	4.1±0.9	4.7±0.9	5.1±0.9	7.5±0.9	9.5±0.9	9.9±0.9
8.0	4.6±0.9	5.1±0.9	5.6±0.9	8.2±0.9	10.4±0.9	10.8±0.9
9.0	5.1±0.9	5.7±0.9	6.1±0.9	8.9±0.9	11.3±0.9	11.6±0.9
10.0	5.7±0.9	6.2±0.9	6.6±0.9	9.6±0.9	12.1±0.9	12.6±0.9

*the error indicated is standard deviation

Table 4. Bulk etch rate and Activation Energy of Makrofol-E and Lexan Polycarbonate detectors

Annealed Temperature	Bulk Etch Rate (μm/hr)					Unannealed	Activation Energy (Kj mol^{-1})
	150ºC	140ºC	120ºC	100ºC	80ºC		
Makrofol-E	0.38	0.425	0.585	0.75	0.835	1.095	9.264
Lexan	0.333	0.482	0.605	0.69	0.77	0.93	9.213

Figure 1. Variation of $Log_{10}V_G$ with $\frac{10^3}{T}$ K^{-1} to find Activation Energy in Lexan and Makrofol-E



5. Conclusion

ISSN: 0971-1260

Vol-22-Issue-17-September-2019

Annealing has an effect on polycarbonate detectors at temperature higher than 120^{o}C. Annealing effect on detector can only be observed when etched for long time. Bulk etch rate for surface etching is same (within error limit) for annealed and unannealed detector when etched for a short time period. To observe significant variation in bulk etch rate the detector has to be etched for longer time period. It can be concluded from our study Makrofol-E detector with density 1.14 gm cm^{-3} has slightly higher bulk etch rate than compared to Lexan polycarbonate detector with density 1.20 gm cm^{-3}, and there is no difference in activation energy for both the polycarbonate detectors.

6. References

[1] S.A. Durani and R.K. Bull, "*Solid State nuclear track detection"*. Pergamon Press, Oxford 1987.

[2] R.L. Fleischer, P.B. Price and R.M. Walker, "*Nuclear Tracks in Solids, Principles and Applications"*, University of California Press, Berkeley 1975.

[3] K.K Dwivedi, "On the critical etching parameters of track detectors"**,** *Radiation measurements,* **27**, 453-456, 1997.

[4] J. P Gewali "*Studies on heavy ion electronic stopping power and thermal fading characteristics of nuclear tracks in solid state nuclear detectors*", Ph. D. Thesis, North-Eastern Hill University, Shillong, India 2014.

[5] J P. Gewali and A. Saxena "Annealing characteristics of 11.56 MeV/u ^{58}Ni irradiated Makrofol-E detector" *Indian Journal of Science and Technology*, **6**, 63-66, 2013.

[6] J. P. Gewali and A. Saxena, Isotropic etching characteristics of Makrofol-E, *Journal of Applied and Fundamental Sciences*, **1**, 81-84, 2015.

ISSN: 0971-1260

Vol-22-Issue-17-September-2019