

Effect of thermal annealing on the maximal apparent etched track lengths of alpha particles in a CR-39 nuclear track detector

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ABSTRACT : In this paper, Poly allyl Diglycol Carbonate (PADC) Polymer-Based Nuclear Track Detector Measurement of Maximum Apparent Track Length of Alpha Particles in NTDs, its brand name is CR-39, has been performed. We determined the etching length of alpha particles from minimum to maximum in an unheat-treated (pristine) and heat-treated CR-39 detector before and after the over-etch zones. The etched alpha track diameter of unheat-treated and heat-treated CR-39 detectors was calculated as a function of etching time at different annealing times and temperatures, using a 241Am as a radioactive source. the energy delivered by the source is 5 MeV which reduced by 0.486 MeV because of 3mm collimator from 5.486 MeV. The resulting minimum and maximum etching lengths for alpha particles in the heat-treated CR-39 detector were smaller than the expected etching lengths, especially at higher annealing temperatures and times. These results are also revealed that the thermally treated CR-39 detectors are insensitive to visualize the real track range of alpha particles at the higher the annealing times and temperatures. The measurements of maximum etched alpha-particle track length are clearly confirmed that the changes brought to the material by heat treatment are chemical in nature not physical, and that these changes significantly alter the measured parameters.

KEYWORDS: CR-39 detectors; CR-39 nuclear track detector; Thermal treatment; Over-etched track diameter method; Alpha particle range means maximum etchable track length; Minimum and maximum etchable track lengths.

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I. INTRODUCTION

The deceleration of the projected ions is caused by inelastic collisions between bound electrons in the medium and ions moving through the medium. The range of a particle corresponds to the distance that the particle travels through the medium until it reaches thermal equilibrium. The range depends on many factors, such as initial energy, particle type, and the material through which the particle passes. It is a one-to-one function of energy for a particular charged particle. Therefore, This range clearly illustrates the particle's kinetic energy for a specific medium and solid charged particles. Therefore, by precisely determining their range, we can accurately determine the energies of these particles. The investigation of how to accurately determine the range of charged particles using nuclear detectors was thus an important goal in the nuclear track community.(R.Fleischeretal,1970) . One of the most interesting methods have been proposed by later Fromm et al (M. Fromm, F. Membrey, 1993), and Almasi and Somogyi.(G. Almási and G. Somogyi, 1981) , To calculate a charged particle incident's range (R) in a nuclear track detector. The track diameter for normally incident charged particle is measured using the proposed over-etched track method as a function of etching time. In actuality, this method is dependent upon the over-etched track's diameter dimension For an over-etched track,

$t \gg t_R$, where t_R is the amount of time needed to etch at speed V_B to the track's end, There is a linear relationship between the square of D^2 diameter and the etching time t . Indicates Experimentally, the range of particles can be determined by calculating the linear regression of the straight-line segment see (A.F. Saad et al , 2023) and references therein for full information. Because it only needs to measure the diameters of the chemically etched track, this method is incredibly easy to use. Even so, It has been successfully used in this situation of slowed-down alpha particles in PADC material (B. Dörschel et al ,1999) and (El Ghazaly et al ,

2012) . Consequently, the nuclear track detectors CR-39 irradiated by different types of light ions such as protons, alpha particles and so on have already been studied by several research groups, as well as the effects on the thermal annealing on these detectors have also been investigated (A.F. Saad, et al ,2012) and (A.F. Saad, et al ,2021)

Finally, because the polymer detector material may have been structurally damaged or chemically altered by heat treatment, the maximum track length of the etching may be equal to the range. If the detector material has been chemically changed, then V_B and V_T should indeed also be altered and consequently the measurements of maximum etched lengths will be affected accordingly. The major goal of the research is to show how the measured maximum etched lengths may account for thermal treatment effects when compared to the non-thermally treated range.

II. EXPERIMENTAL PROCEDURE

In this study, CR-39 detection sheets with a thickness of 750 μm were acquired from Track Analysis Systems Ltd. (TASL) in Bristol, UK. Using a laser beam, the sheets were precisely divided into uniform pieces measuring $1.5 \times 1.5 \text{ cm}^2$ each. The studied samples were divided into two sets; These two sets included those with heat treatment and α particles (post-annealing exposure) and vice versa (pre-annealing exposure) compared to the non-heat-treated sample (pristine sample) as a control. To ensure a normal α -particle ratio and avoid contamination of the detector, we used a 3 mm diameter collimator that reduces the energy from 5.48 to 5.0 MeV (A.F. Saad, et al ,2019) and the contained reference. The specimens were exposed to 5.0 MeV α -particles emanating from a ^{241}Am source. Additionally, thermal annealing procedures were executed at temperatures of 100, 110, and 120 $^\circ\text{C}$, each spanning a duration of 6 h using a monitored oven. The oven is manufactured by Binder Company (Model:FP 720), Germany and can operate at temperatures from 5 $^\circ\text{C}$ above ambient temperature to 300 $^\circ\text{C}$. After heat treatment and irradiation, the detectors were then etched in a standard 7.25 N NaOH aqueous solution maintained at 70 $^\circ\text{C}$ by water bath, representing the most traditional etching conditions. for CR-39 NTD. The temperature is upheld consistently with a maximum error of ± 1 $^\circ\text{C}$. The etching duration was selected to enable the visibility of the alpha trace under an optical microscope, magnified at 640 times. For the current experiments, Four CR-39 detector samples were employed, comprising identical detector models as previously described for each rate measurement through the weight loss method. The presented data encompasses information from three annealing cycles and results from 106 etching cycles. Fifty circular trace were utilized to assess the diameter of the α -particle traces. These traces exhibited nearly identical major and minor axes and were notably darker compared to the remaining traces.

III. RESULTS

Higher isochronal annealing experiments often result in significant polymer degradation. The change in polymer chain spacing during annealing also changes the physical and chemical properties of the PADC polymer, changing the detection threshold of the affected polymers. The PADC material becomes significantly softer, resulting in increased the bulk etch rate and decreased detection efficiency. Table 1 shows the values of degree of softening (DS %),

which is the increase in bulk etch rate in percentage at etching duration of 2 h for CR-39 detector material. The values of degree of softening vary from 7.3, 14.6 and 154.5 % corresponding to the annealing temperatures of 100, 110 and 120 $^\circ\text{C}$ and annealing time 6 h. The surface material of the PADC film is permanently softened, resulting in an increase in the bulk etch rate, which represents the degree of softening, and a decrease in the detection sensitivity and detection threshold of α particles, as will be discussed in the forthcoming items.

The measurements of etched alpha-particle track lengths, overall etching duration times from 2 to 24 hours or 8 hours for highest annealing temperature, in non-heat treatment (pristine) sample, see Table 2,

Table 1

Values of degree of softening of pristine and thermally treated CR-39 detectors at different annealing temperatures for a duration of 6 h.

Annealing temp. (°C)	Annealing time (h)	Degree of softening (%)	
		Un-annealed	Exposure post- annealing
Pristine	-	-----	-
100	6		7.3
110	6		14.3
120	6		154.5

Table 2

Values of bulk and track etch rates as well as etchable track lengths at etching time ranging from 2 to 24 h in a non-annealed (pristine sample) CR-39 nuclear track detector. The etching was performed until the alpha tracks were reached the over-etched stage and beyond the over-etched zone.

Etching Time (t)	Bulk etch rate (V_b)	track etch rate (V_T)	Track length (L_a)
2	2.28	3.19	1.82
4	2.3	3.24	3.76
6	2.31	3.52	7.26
8	2.33	3.55	9.76
10	2.29	3.57	12.8
12	2.31	3.65	16.08
14	2.31	3.65	18.76
16	2.3	3.7	22.4
18	2.32	3.71	25.02
20	2.3	3.72	28.4†
22	2.34	3.79	31.9‡
24	2.33	3.78	34.8

† Stands for the alpha tracks before the over-etched stage.

‡ Stands for the alpha tracks after the over-etched stage.

and heated treatment CR-39 detectors, see Tables 3-5, were performed. We found that the changes made to the material by heat treatment are chemical rather than physical in nature and that these changes significantly alter the measured parameters.

Table 3

Values of bulk and track etch rates as well as etchable track lengths at etching time ranging from 2 to 24 h in an annealed CR-39 nuclear track detector. The post-annealing was performed at temperature 100 °C for duration time of 6 h. The etching was performed until the alpha tracks were reached the over-etched stage and beyond the over-etched zone.

Etching Time (t)	Bulk etch rate (V_b)	track etch rate (V_T)	Track length (L_e)
2	3.20	5.0	3.6
4	2.80	5.0	9.0
6	2.65	5.7	18.6 †
8	2.77	5.2	19.1 ‡
10	2.61	4.9	23.8
12	2.78	4.9	25.2
14	2.63	4.8	30.5
16	2.71	4.8	30.9
18	2.69	4.7	36.1
20	2.68	4.6	37.5
22	2.68	4.4	38.8
24	2.68	4.3	38.7

† Stands for the alpha tracks before the over-etched stage.

‡ Stands for the alpha tracks after the over-etched stage.

Table 4

Values of bulk and track etch rates as well as etchable track lengths at etching time ranging from 2 to 24 h in an annealed CR-39 nuclear track detector. The post-annealing was performed at temperature 110 °C for duration time of 6 h. The etching was performed until the alpha tracks were reached the over-etched stage and beyond the over-etched zone.

Etching Time (t)	Bulk etch rate (V_b)	track etch rate (V_T)	Track length (L_e)
2	2.91	8.5	11.1 †
4	2.86	9.2	25.5 ‡
6	2.90	10.1	43.4
8	2.90	9.2	50.5
10	2.87	7.8	49.2
12	2.87	7.5	55.1
14	2.86	7.1	58.9
16	2.83	6.6	59.5
18	2.78	6.4	65.5
20	2.78	6.1	66.7
22	2.78	5.7	65.3
24	2.76	5.5	66.8

† Stands for the alpha tracks before the over-etched stage.

‡ Stands for the alpha tracks after the over-etched stage.

Table 5

Values of bulk and track etch rates as well as etchable track lengths at etching time ranging from 2 to 8 h in an annealed CR-39 nuclear track detector. The post-annealing was performed at temperature 120 °C for duration time of 6 h. The etching was performed until the alpha tracks were reached the over-etched stage and beyond the over-etched zone.

Etching Time (t)	Bulk etch rate (V_b)	track etch rate (V_T)	Track length (L_e)
2	2.91	8.5	11.1 †
4	2.86	9.2	25.5 ‡
6	2.90	10.1	43.4
8	2.90	9.2	50.5
10	2.87	7.8	49.2
12	2.87	7.5	55.1
14	2.86	7.1	58.9
16	2.83	6.6	59.5
18	2.78	6.4	65.5
20	2.78	6.1	66.7
22	2.78	5.7	65.3
24	2.76	5.5	66.8

† Stands for the alpha tracks before the over-etched stage.

‡ Stands for the alpha tracks after the over-etched stage.

Table 6

Range of alpha particles in the non-thermally treated (pristine sample) and maximum etched track length in the thermally treated CR-39 detectors at a boarder of over-etched zone. Energy of alpha particles 5.454 MeV is reduced to 5.0 MeV using 3-mm collimator. Thermal annealing at different annealing temperatures for a 6 h duration time.

Annealing temp. (°C)	Annealing time (h)	Range or maximum etched track lengths (μm)	
		Un-annealed	Exposure post annealing
Pristine	-	29.6 ± 0.9	-
100	6		18.9 ± 0.9
110	6		14.8 ± 0.3
120	6		11.7 ± 0.5

The etched track diameter method, before and after over-etched area, was applied to measure the particle's etched length at 5 MeV energy in a non-heat treatment (pristine) and heat treatment sample (exposure post-heat treatment) CR-39 detector for various annealing temperatures of 100, 110 and 120 °C at an incubation period of 6h. After the end of the alpha track, track diameter measurements were taken as a function of etching time. Photomicrographs of the etch pits caused by alpha particles incident on non-heated treatment and heated treatment CR-39 samples at 100 and 120 oC are presented as a function of etching times for two modes over a period of 6 hours; pristine (see Fig. 1)

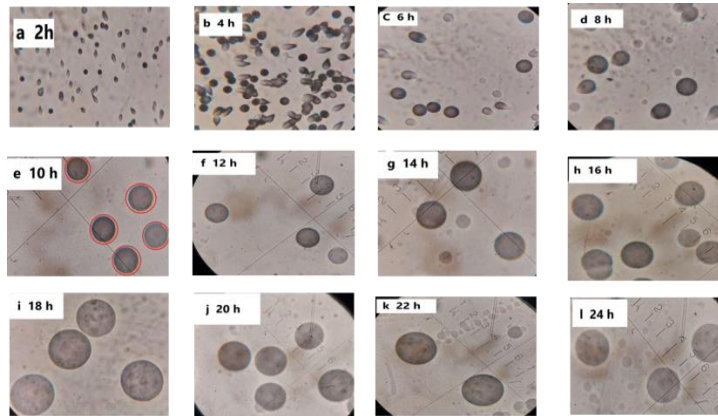


Fig.1. Photomicrographs of etch pits due to alpha particles incident on non-thermally treated CR-39 detector for etching duration periods from 2 to 36 h. ²⁴¹Am exposure was performed on pristine CR-39 sample.

exposure post-heat treatment (see Figs. 2-3). The relationship between etching time of exposure post-heat treatment and the squared diameter of the α -particle track for annealing temperatures 100, 110 and 120 °C at annealing time interval of 6 h has been carried out, in addition to the pristine sample.

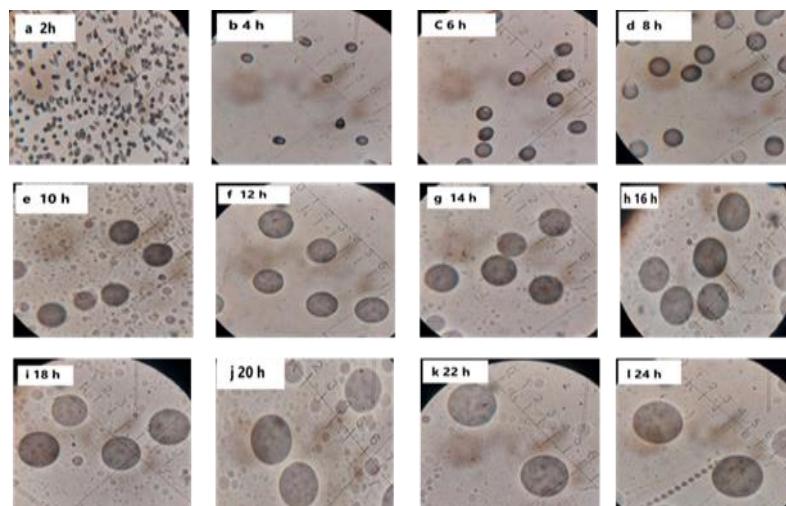


Fig. 2. Photomicrographs of etch pits due to alpha particles incident on annealed CR-39 sample at 100 °C for duration period of 6 h. Etching was carried out from 2 to 36 h. ²⁴¹Am exposure was performed post-heating

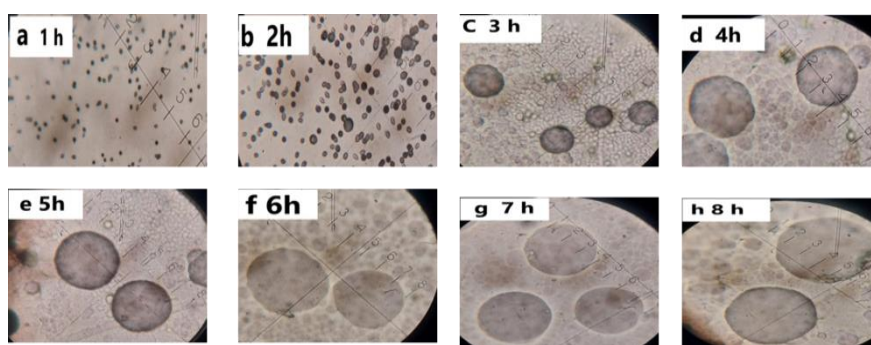


Fig. 3. Photomicrographs of etch pits due to alpha particles incident on annealed CR-39 sample at 120 °C for duration period of 6 h. Etching was carried out from 2 to 36 h. 241Am exposure was performed pre-heating

As an example, The relationship between the square diameter of the α -particle trace and etching time has been presented for pristine, post-annealing exposures at annealing temperatures of 100 and 120 °C for 6 h for comparison, as shown by shown in Fig.4, After obtaining the parameters S and I, Rexp can be calculated using equation (3). as detailed (A.F. Saad et al , 2023).

Table 6 shows the experimental ranges Rexp of α -particle at 5 MeV for the pristine sample and those exposed to the heat treatment modes. When compared to the pristine sample, Rexp values in the second mode (post-annealing exposure) showed a clear decrease of up to 36.1% over a 6-hour annealing time interval at a temperature of 100 °C. Whereas By increasing the temperature by 10°C to reach 110°C under the same conditions, we found that Rexp decreased even further to reach 50.0%. Similarly, at 120°C, Rexp also has a significant decrease, reaching a value of 60.5%.

The exposure mode following annealing led to a sustained reduction, surpassing 50% or more than half of the original sample value, achieved by elevating the annealing temperature to 120°C over a 6-hour annealing period. The α -particle range derived from 241Am in a CR-39 sample without thermal treatment (pristine sample), measuring 29.6 μm , exhibits notable consistency with the findings in our recent study, both experimentally and theoretically. (A.F. Saad et al , 2023)and references therein.

Indeed, the etching length of α -particles shows a clear decrease in the heat-treated CR-39 detector compared to the pristine sample, indicates that the length of the region damaged by α particles has chemically etched (the latent trace zone), significantly reduced at higher temperatures and longer time treatment. In fact, there is no reason why the range of alpha particles in the detector material should be changed by heat treatment. The reason for this change is that the physical properties of the material interaction cannot be modified by simple chemical modifications of the medium resulting from heat treatment. The range of an alpha particle in a medium, and therefore its linear energy transfer, is influenced by the medium's average ionization potential and electron density. These factors aren't predicted to be highly sensitive to structural or chemical changes in the medium caused by such a large amount of heat treatment. As a result, thermal treatment significantly alters the entire material chemistry. (A.F. Saad, et al ,2012) and (A.F. Saad, et al ,2021).leading to a rise in VB and, as a result, a visible decrease in the maximum measurable etchable length (range) Rexp (A.F. Saad, et al ,2023)This is demonstrated in Table 6, where a notable increase in the chemical etching rate can be seen in the case of heat treatment at 120°C, as shown in Table 5.

IV. Conclusion

We have determined the maximal apparent etchable lengths of α -particles by analysing before and after diameters of over-etched tracks in non-heated treatment and heated treatment CR-39 nuclear track detector over period of 6 h, at three different temperatures. The results propose that The thermal dose determines how much the apparent decreasing particle range is altered by heat treatment. Exposure post-heat treatment has shown to be a unique method in the present work , causing constant, ongoing decreases in the apparent α -particle range values. This study represents the initial investigation reporting on the determination of alpha-particle etchable length in a CR-39 nuclear track detector following exposure in a post-heat treatment mode.

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