Indian Journal of Pure & Applied Physics
Vol. 61, June 2023, pp. 504-508
DOI: 10.56042/ijpap.v61i6.2430

A Study on the Bulk Etch Rate of LR-115 Solid State Nuclear Track Detector using Lithium Hydroxide as an Etching Solution

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Received 20 February 2023; accepted 23 May 2023

Among numerous applications of Solid-State Nuclear Track Detector (SSNTD), one important application is estimating radon and thoron concentration in an indoor environment. The primarily used SSNTD for this application is LR-115. Studying the behaviour of these SSNTDs for different etching solutions is essential. In this report, Lithium hydroxide solution was used to etch LR-115 SSNTD. The study of the thickness of the film removed is carried out at different normality of etching solution to study the effect of normality of etching solution on the bulk etch rate. The change in bulk etching rate with the change in temperature of the etching solution was also put out in this study. From the results, it is discovered that the bulk etch rate is directly proportional to the normality and temperature of the etching solution. Additionally, it can be deduced that the LiOH bulk etching rate of LR-115 has lower values when compared to those of other alkaline hydroxide etching solutions.

Keywords: Bulk etch rate; Normality; Temperature; LR-115, Lithium hydroxide

1 Introduction

The detection of radionuclides using a plastic detector was observed when Young discovered fission fragments pits in lithium fluoride[1]. Many dielectric materials, such as solids, minerals, glasses, polymers, *etc*., were employed to detect heavily charged particles. The primary detection principle of these detectors is the detection of individual tracks formed in these detectors when heavy charge particles pass through. Solid State Nuclear Track Detectors (SSNTD) are immensely employed for detecting indoor radon and thoron concentration by researchers all around the world[2-5]. The two mainly employed SSNTDs for this purpose are CR-39 and LR-115. The characteristic property of this detector is very high insensitivity to electron, neutron, proton and gamma radiations[6]. LR-115 is composed of cellulose nitrate dielectric plastic and is developed by DOSIRAD, France[7]. It is composed of two layers, one of cellulose nitrate film of thickness 12 µm layer and a 100 µm thick polyester base. The cellulose nitrate film is used to form tracks of heavily charged particles. After the formation of tracks, delineating tracks formed by heavy charged particles is essential to calculate the track density precisely. For this delineation process, many methods are used, such as chemical etching, electrochemical etching, and polymer grafting[8-9], but the most suited method is chemical etching. In this method, the SSNTD were submerged in a solution of fitted chemical reagents capable of etching some of the tracks formed (mostly hydroxyl of alkaline metals). However, this etching technique has many factors that can affect the etching process, like temperature and concentration of the etching solution, the etching solution used, the configuration of the films and some impurities. Studying these factors is crucial in calculating the density of tracks formed in these detectors. In this report, variation of etching with variation in temperature, concentration and etching reagent is studied.

Two essential parameters in the etching of these SSNTD are the etching rate of the tracks formed, and the surface etching rate. Track etch rate is calculated to know the increase in the size of tracks per unit of time, and bulk etching rate can be defined as the thickness of film etched per unit of time. This paper presents the variation of bulk etch rate of LR-115 SSNTD at different temperatures and the normality of etchant. The bulk etching rate depends upon various factors like chemical composition, structure, etching reagent, etching conditions, stirring, external irradiation[10] *etc*. To calculate the bulk etch rate, the

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thickness of these films at different time intervals of the etching is an important parameter. Many methods, such as direct measurement[11], gravimetric method[12], PHEONIX interface[13], FTIR Spectroscopy[14] *etc*., are used by researchers worldwide to calculate the removed thickness of the cellulose nitrate layer. The detection principles of all these techniques are different. Here, the thickness of the removed active layer was calculated using direct and gravimetric methods.

In the gravimetric method, the thickness of the active layer removed is calculated by calculating the percentage mass change before and after the etching of films. Also, the results of both methods were compared to check the accuracy of the gravimetric method by comparing it with direct results.

2 Experiment Details

The SSNTD used in this study is an LR-115 type-2 strippable detector composed of a 100 µm polyester base and a 12 µm active layer of cellulose nitrate with a total thickness of 112 µm[12]as shown in Fig. 1.

Six films of equal size (2 cm ×2 cm) were cut and etched at different periods of 15 min, 30 min, 45 min, 60 min, 75 min, and 90 min at a particular normality and temperature of the etching solution. This procedure was then repeated by changing either the normality or temperature of the etching solution.

To estimate the variation of bulk etch rate (V_{bulk}) of LR-115 SSNTD with the normality of LiOH solution, the bulk etch rate is calculated at 1.0 N, 1.5 N, 2.0 N and 2.5 N normality of lithium hydroxide solution at a constant temperature of 60 °C. Similarly, to analyze the variation of bulk etch rate with the temperature of etching solution, the bulk etch rate is calculated at different temperatures viz., 50°C, 55 °C, 60 °C, 65 °C, 70 °C at constant normality of 2.5N of lithium hydroxide solution. A constant-temperature water bath developed by Polltech Pvt. Ltd., is used to maintain a constant temperature throughout the experiment. The bulk etching rate calculation was done by plotting the graph between the thickness of the active layer removed and the time of etching. The slope of this graph was then used to calculate the bulk tech rate. The bulk etching rate in terms of thickness removed in µm per unit hour of etching time.

Fig. 1 — Basic schematics of LR-115 SSNTD.

Two methods were deployed to measure the thickness of SSNTD removed at different time periods, *i.e*., direct and gravimetric methods. The direct method is based on the direct measurement of the thickness of the active layer removed. In contrast, in the gravimetric method, the removed thickness is calculated by measuring the percentage change in mass of the films before and after the etching.

2.1 Direct method

In this method, we measure the removed thickness of the active layer directly by using the digital micrometer by Mitutoyo.

This device has a pitch of about 125 mm with the least count of 1 µm. The removed thickness can be directly analyzed by subtracting the thickness of the films after etching from the thickness before etching. This method is used by Mehta et al. (2015)[10]to estimate the bulk etch rate.

2.2 Gravimetric Method

In this method, the bulk etch rate of LR-115 films is calculated by calculating the mass of films before and after the etching. As we know, mass = density/ volume or

$$m = \rho \times a \times t$$

Here a is the area of films, and t is the thickness. By considering that area does not change with the etching, we can write the following equation

$$M_{initial} = a\,(\rho_{pol} \times t_{poly} + \rho_{active\ layer} \times t_{active\ layer\ initial}) \quad (1)$$

$$M_{final} = a\,(\rho_{pol} \times t_{poly} + \rho_{active\ layer} \times t_{active\ layer\ final}) \quad (2)$$

Here ρ_{pol} is the density of the polyester base, t_{poly} is the thickness of the polyester base, $\rho_{active\ layer}$ is the density of the cellulose nitrate active layer, $t_{active\ layer\ initial}$ is the thickness of the active layer before etching, and $t_{active\ layer\ final}$ is the active layer thickness after etching.

By redistributing equations (1) and (2), we can get the equation of change in thickness as follows.

$$\Delta t = \left(t_{active\ layer\ initial} + \frac{\rho_{pol}}{\rho_{active\ layer}} \times t_{poly}\right)\left(1 - \frac{M_{final}}{M_{Initial}}\right) \quad (3)$$

So, by substituting the values of the density of polyester as 1.395 g/cm^3, the density of cellulose

nitrate as 1.44 g/cm^3, and the thickness of polyester as 100 μm in equation (3), we got

$$\Delta t = 108.875 \times \left[1 - \frac{M_{final}}{M_{Initial}}\right] \quad (4)$$

So, by knowing the initial and final mass of the LR-115, the thickness removed can be calculated using equation (4). A digital weighing balance by Wensar with the least count of 0.001 g is used for measuring mass precisely.

3 Results

This paper presents the effect of change in normality and temperature of lithium hydroxide etching solution on the bulk etch rate of Lr-115 SSNTD. The variation of surface chemical etching with the normality of the etching solution used is studied by measurement of thickness removed at the normality of 1.0 N, 1.5 N, 2.0 N, 2.5 N etched at a constant temperature of 60 °C. The removed thickness of SSNTD is calculated at different normality using the method viz., direct method and gravimetric method. The results of bulk etch rates at different normality are given in Table 1. It can be seen from the results shown in the table that the bulk etch rate increases with an increase in the normality of lithium hydroxide solution. A gradual increase is discovered in the normality of the etching solution.

Also, comparing the results of the bulk etch rate of the direct method with the gravimetric method, we discovered that the results by both methods are approximately the same.

The graph of the bulk etch rate calculated at different normalities calculated using the direct and gravimetric methods is also plotted and shown in Fig. 2. The graph shows a linear increase in bulk etch rate with an increase in the normality of the etching solution. The slope of the graph was found to have a value of 1.03 (μm/h)/N.

Similarly, the bulk etch rate variation is also calculated with the variation in temperature of the etching solution of LiOH. For this purpose, the films were etched for different periods for different etching temperatures of about 50 °C, 55 °C, 60 °C, 65 °C, and 70 °C at a constant normality of 2.5 N of lithium hydroxide etchant. Both methods were used to calculate the V_{bulk}. For each temperature, the graph was plotted between the period of etching and thickness of the active layer removed, and V_{bulk} was then calculated by calculating the slope of bulk etch rate vs time graph. The results of the calculated bulk etch rate at different temperature using direct and gravimetric method is given in Table 2.The results showed that the bulk etch rate increases with the temperature of the etching solution.

The scatter plot between the bulk etch rate and temperature of the etching solution is also plotted and is shown in Fig. 3.The graph is plotted to check

The variation of bulk etch rate with the increase in the temperature of the etching solution. We discover from the graph that though there is an increase in bulk etch rate on increasing the temperature, there is not a linear increase as in the case of normality. Instead, an exponential increase was discovered by increasing the temperature of the etching solution.

Similar results were also quoted in other reports, such as Duranni *et al*., (1982), Tse et al., (2007), and Mehta *et al*., (2014) [16-18]but the etching solution used

Table 1 — Bulk etch rate at different normality using both methods

Normality	Bulk Etch Rate (μm/h)	
	Direct Method	Gravimetric method
1.0 N	1.8 ± 0.11	1.92 ± 0.12
1.5 N	2.15 ± 0.14	2.25 ± 0.18
2.0 N	2.94 ± 0.16	3.04 ± 0.19
2.5 N	3.34±0.18	3.45 ± 0.19

Table 2 — Bulk etch rate at different temperature of etching solution calculated by direct and gravimetric methods

Temp. (°C)	Direct method (μm/h)	Gravimetric Method (μm/h)
50	2.8±0.122	2.9±0.16
55	4.14±0.93	4.24±0.79
60	4.89±0.178	4.76±0.188
65	6.2±0.208	6.4±0.221
70	7.49±0.498	7.61±0.166

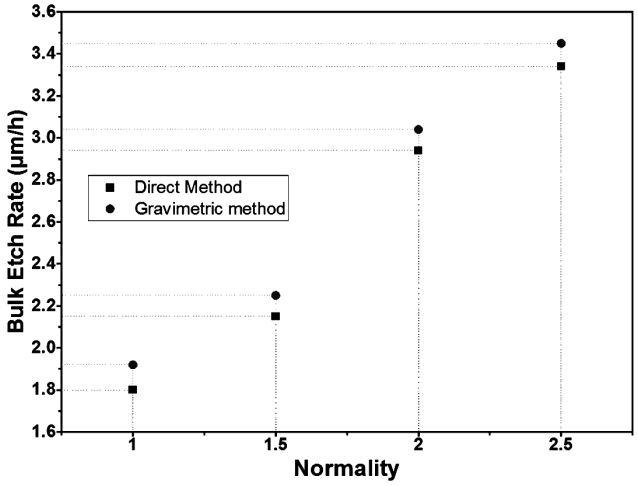

Fig. 2 — Graph between normality and bulk etch rate calculated using direct and gravimetric methods.

was sodium hydroxide. To compare the different etching solutions, a comparison experiment was performed by having the same etching condition with a different etching solution. This study's results are shown in Fig. 4. In this plot, the values of bulk etch rate at different temperature values are shown by scatter plot.

Potassium hydroxide has the highest bulk etch rate among LiOH and NaOH due to its low activation energy. KOH is more basic and dissolves more easily in water than LiOH and NaOH, which explains why it has a higher bulk etch rate. The stronger base of KOH allows for a more rapid reaction with the etched material, and its higher solubility enables deeper and more thorough etching. As a result, KOH exhibits a greater etch rate than NaOH and LiOH. The difference in bulk etch rates between lithium hydroxide (LiOH) and sodium hydroxide (NaOH) is also noticeable, with LiOH requiring significantly longer etching times to achieve the same amount of etching as NaOH. For instance, under 2.5 N and 60 °C etching conditions, NaOH reached the desired etching in just 90 minutes, while LiOH required a much longer etching time of 110 minutes. Given these extended etching times and slower etch rates, LiOH is not a preferred choice for the etching of SSNTD films.

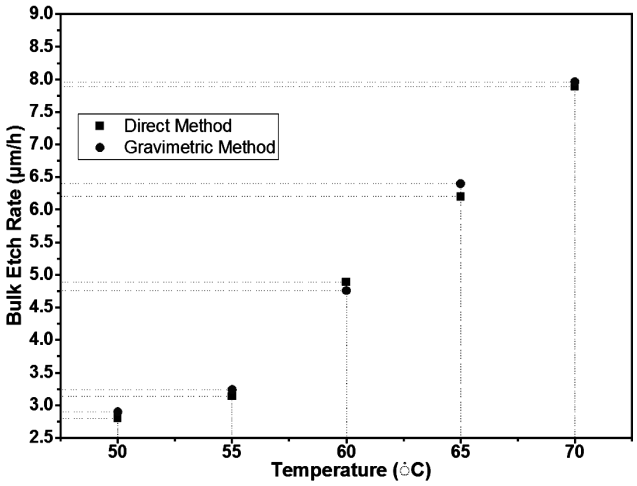

Fig. 3 — Graph between temperature and bulk etch rate calculated using direct and gravimetric methods.

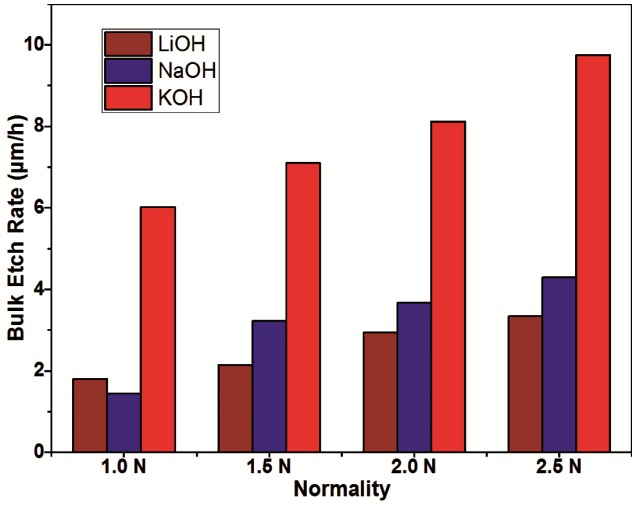

Fig. 4 — Graph showing the bulk etch rate of KOH, NaOH and LiOH at different concentrations.

4 Conclusions

Following conclusions can be drawn from the above results.

1. As the concentration of the lithium hydroxide etching solution is increased, the bulk etch rate of the LR-115 SSNTD increases linearly.
2. The change in bulk etch rate with temperature may be seen in the fact that as the temperature of the lithium hydroxide etching solution rises, so do the bulk etch rates. The increase is not linear, though, and substantial increases have been found at higher temperatures.
3. Comparing the bulk etch rate of LR-115 SSNTD with KOH, LiOH, and NaOH revealed that potassium hydroxide had the highest etch rates while lithium hydroxide had the lowest etch rates.

Acknowledgement

The authors like to thank Dr. Jasvir Singh, Principal. Sri Guru Teg Bahadur Khalsa College, Sri Anandpur Sahib, for facilitating this research work. This work is supported under BRNS- DAE funded research project (Sanction No.:56/14/01/2019-BRNS) and DST-FIST Scheme (No. SR/FST/College-2019-759(C)).

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