# A Study on Surface Chemical Behaviour of Solid State Nuclear Track Detector Films by Etching

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**Abstract:** *The etching is a process of modifying the flat smooth surface to identify the structure, phases, and other effects such as the orientation of grains, deformation and distribution of solute elements. Due to the chemical reaction between the etchant and the detector material, some molecules of the detectors are removed. The etchant used for LR-115 detectors in the present study is aqueous NaOH solution with 2.5 N. The bulk etch rate has been estimated by varying the etching time, keeping the etching temperature constant. It has seen that, the bulk etch rate remains almost constant during etching. The result indicates that, rate of chemical etching is uniform in different intervals of etching time. The average bulk etch rate obtained from experiments was in good agreement with the standard value obtained from earlier experiments.*

**Key Words:** *LR-115, Etching, Bulk Etch Rate.*

## Introduction

Cellulose nitrate ($C_6$-$H_8O_8$-$N_2$) films (commercially available as LR-115 films from DOSIRAD, France) have been commonly used as solid state nuclear track detectors (SSNTDs) in which visible tracks can be formed after ion irradiation and suitable chemical etching. SSNTDs are passive devices since these devices require no electrical power to perform its function. The films are mainly intended for the dosimetry of small quantities of ionizing particles (mainly "alpha particles") or neutrons and are insensitive to beta and gamma rays. These are well known for the detection of ionizing radiation through track formation of heavy ionizing particles and are used to measure $^{222}$Rn, $^{220}$Rn and their progenies. The exposed SSNTD are required to undergo an etching process which turns the nuclear tracks on the films into holes. For the analytical results to be reliable and precise, temperature and concentration of the etching solution and etching time have to be stable and controlled. Constant Temperature Bath Model PSI-CTB1 is excellent equipment which facilitates processing of up to 30 films at one go.

Ion track growth in SSNTDs has been suggested to be based on two parameters, $V_t$ and $V_b$ where, $V_t$ is the track etch rate (i.e., the rate of chemical etching along the ion trajectory) and $V_b$ is the bulk etch rate (i.e., the rate of chemical etching of the undamaged surface) (Yip et al., 2006). The second factor $V_b$ is strongly related to the removed thickness of the active layer and it has been established that the thickness of the removed layer during etching of the solid state nuclear track detector is one of the main factors influencing the track parameters or shape characteristics (Hussain, 2009). $V_b$ is one of the most important parameters that control the formation and development of tracks and $V_t$ is needed to simulate track growth and to calculate the track parameters. It has been shown that $V_b$ depends on many factors like the purity of the basic substances, the molecular structures of polymers, conditions of polymerization, environmental conditions during the irradiation and finally on etching conditions (Palacios et al., 2010). $V_b$ can be calculated using several methods, such as the decrease in the detector's thickness method, the loss of the detector's weight method or the fission-fragment diameter method (Hassan and Hafez, 2013).

The LR-115 type II SSNTD is used in the present study. The detector consists of a 12μm active layer thickness of cellulose nitrate on a 100μm clear polyester base substrate. Mass difference method is used to determine the bulk etch rate ($V_b$) and the removed active layer thickness (h) of LR-115 type II detectors.

## Materials and Methods

The LR-115 type II strippable detectors used in the present study were purchased from DOSIRAD, France (supplied by Polltech Instrumentation Pvt. Ltd., Mumbai). The detectors consist of a nominal 12 μm thick red strippable active layer of red cellulose nitrate on a 100 μm clear polyester base substrate. The Constant Temperature Bath Model PSI-CTB1 used in the present study, comprises a double-walled water bath with glass wool insulation, three etching vessels, electrical water heater, circulating pump, temperature sensor and electronics. Each of the three etching vessels has slots for a rod that can accommodate 10 cartridges to facilitate the processing up to 30 films at one time. The electrical water heater is a resistive type heater. Circulating pump is used to circulate the water during the heating process to ensure uniform heating.

The etching is the process of modifying the flat smooth surface to identify the structure, phases, and other effects such as the orientation of grains, deformation and distribution of solute elements. Due to the chemical reaction between the etching solution (etchant) and the detector material, some molecules of the detectors are removed. Aqueous NaOH solution with 2.5 N (10%) is the commonly used etchant for LR-115 detectors.

During the present work, bulk etch rate has been estimated by varying the etching time keeping the standard etching temperature (60°C) as constant. The un-irradiated LR-115 detectors were cut with a size of about 3×3 $cm^2$ and their weights were measured using a weighing balance machine of 0.1mg accuracy. After the weight measurement, each detector was dipped in 2.5N (10%) NaOH solution and etching was carried out using the constant temperature bath. The temperatures were kept with an accuracy of about ±1°C. For each etching time, at constant temperature 60°C, the films were immediately rinsed by distilled water and air-dried for 30 minutes.

After completing the etching at various temperatures, the weights of each film were again measured and average mass differences Δm before and after etching were calculated. Because of the non-uniformity in the expected area (9 $cm^2$) of the film, the actual area of the films was calculated using a travelling microscope. Travelling microscope is focused at the surface ends of a film and measured the length and breadth of the film by adjusting the horizontal and vertical cross wire. From this, the area of each film was calculated.

The bulk etch rate was calculated using the equation, $$V_b = \frac{1}{2}\frac{m}{A\rho t} \quad (3.1)$$

where $V_b$ is the bulk etch rate in μm $h^{-1}$, Δm is the mass difference in mg before and after etching, A is the etched surface area in $cm^2$, ρ is the density of the active layer, which is equal to 1.45gm $cm^{-3}$ for LR-115 and t is the etching period in hours.

After calculating the bulk etch rate ($V_b$), it is possible to evaluate the removed-active layer thickness h (μm) at any etching time using the following relation (Hussain, 2009)

$$h = V_b \times t \quad (3.2)$$

## Results and Discussion

Experimental results of the chemical etching of LR-115 type-II SSNTD films at different etching time are given in Table 1. Graph of bulk etch rate Vs. etching time; and removed layer thickness Vs. etching time was plotted and shown in Fig. 1 and Fig. 2 respectively. It can be seen that, the bulk etch rate remains almost constant during etching (Fig. 1). The result indicates that, rate of chemical etching is uniform in different intervals of etching time selected for the experiment.

Thickness of the removed layer is an important factor in all applications of α particle detectors. In the present study, the estimated removed layer thickness shows linear variation against etching time (Fig. 2) at constant etching temperature.

**Table 1: Bulk Etch Rate and Removed Active Layer Thickness**

| Time (minutes) | Area of the film ($cm^2$) | Mass before etching(mg) | Mass after etching(mg) | Mass difference Δm(mg) | Bulk etch rate $V_b$ (μm $h^{-1}$) | Removed layer thickness h (μm) |
|---|---|---|---|---|---|---|
| 60 | 9.4064 | 0.1452 | 0.1384 | 0.0068 | 4.9856 | 4.9856 |
| 70 | 8.999 | 0.1389 | 0.1319 | 0.0070 | 4.5982 | 5.3646 |
| 80 | 9.4885 | 0.1464 | 0.1377 | 0.0087 | 4.7426 | 6.3234 |
| 90 | 8.9287 | 0.1378 | 0.1291 | 0.0087 | 4.4799 | 6.7199 |
| 100 | 8.9559 | 0.1382 | 0.1285 | 0.0097 | 4.4817 | 7.4696 |
| 110 | 9.4444 | 0.1458 | 0.1350 | 0.0108 | 4.3017 | 7.8864 |
| 120 | 9.1506 | 0.1412 | 0.1303 | 0.0109 | 4.1075 | 8.2150 |

The average bulk etch rate obtained from the two sets of experiments was 4.48 μm $h^{-1}$ without stirring and it was in agreement with the standard value obtained from the earlier experiments and was 4 μm $h^{-1}$ (Eappen and Mayya, 2004). The standard etching conditions for the above experiment was constant etching temperature of 60°C without magnetic or any other stirring for 90 minutes etching time. The accuracy of calculation depends on the accuracy of measurement of mass difference for the LR-115 detector.

**Fig. 1: Bulk Etch Rate Vs. Etching Time**

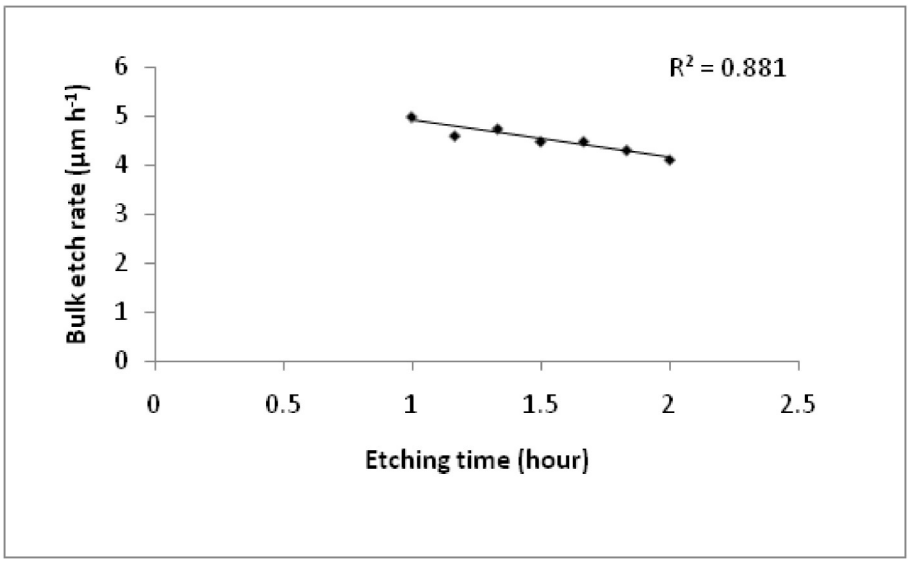
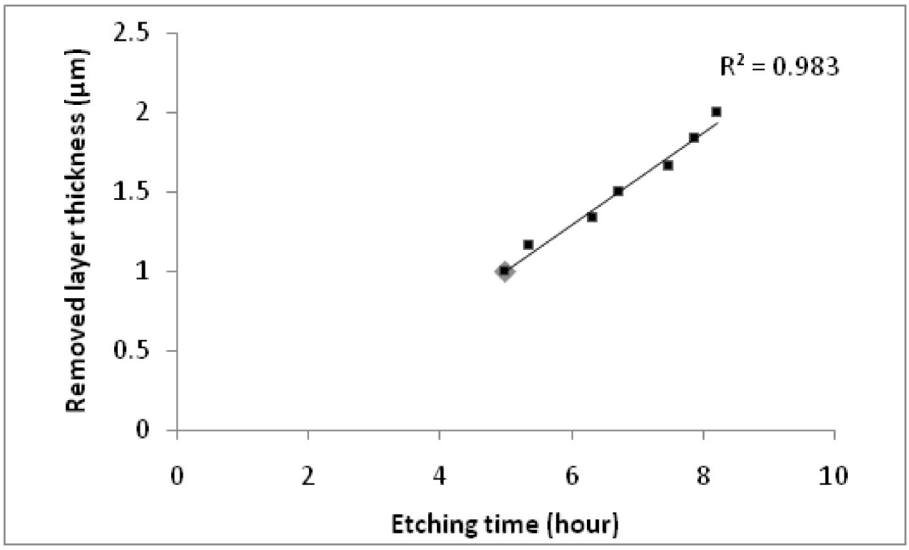



**Fig. 2: Removed Layer Thickness Vs. Etching Time**



## Conclusion

The bulk etch rate estimation was carried out keeping the etching temperature constant and found that, the bulk etch rate remains almost constant during etching. The study also indicates that, rate of chemical etching is uniform in different intervals of etching time. The estimated removed layer thickness showed a linear variation against etching time. In order to conclude, the accuracy in the measurement of mass difference is an important parameter while calculating the various etching parameters of an LR-115 SSNTD films.

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