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SURFACE CHEMICAL ETCHING BEHAVIOR OF LR-115 TYPE II SOLID STATE NUCLEAR TRACK DETECTOR

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Abstract. Solid State Nuclear Track Detectors (SSNTDs) are well known for the detection of ionizing radiation through track formation of heavy ionizing particles. The LR-115 detector is a commonly used SSNTD for the detection and measurement of Rn^{222} and its progeny. Several techniques of track revelation are known but, the chemical etching technique is the most frequently used in which tracks can be made visible under an optical microscope after chemical amplification *via* etching. So the surface chemical etching behavior of LR-115 polymer is an important parameter in the study of ionizing radiation. The surface chemical behavior is studied by etching process. The parameter studied is the thickness of polymeric sample for different time intervals at different temperatures and normality. The results are indicated in terms of bulk etch rate and an activation energy is determined.

Key words: LR-115, chemical etching, bulk etching rate, activation energy.

1. INTRODUCTION

Solid State Nuclear Track Detectors (SSNTDs) are well known for the detection of ionizing radiation through track formation of heavy ionizing particles. The composition, processing, and applications of solid-state nuclear track detectors (SSNTDs) are sufficiently discussed by many researchers [1–4]. SSNTDs, particularly Cr-39 and LR-115, are used to measure the Rn^{222} and its progeny [5–9]. Radon (^{222}Rn) is a radioactive gas produced as a decay product of radium (^{226}Ra) [10]. The red LR-115 type II detector (from DOSIRAD) is based on cellulose nitrate and is a commonly used solid-state nuclear track detector (SSNTD). The LR-115 type II detector is a commercially available red cellulose nitrate detector with an active layer of 12 μm on a 100 μm clear polyester base (as declared by the manufacturer). Alpha particles emitted by Rn^{222} and its progeny hit the detector and leave the latent tracks in it. Several different techniques of track revelation are known, *e.g.* grafting [4, 11] but, the chemical etching technique is the most frequently used in which tracks can be made visible under the optical microscope after chemical amplification *via* etching. Track development in an SSNTD is based on two parameters V_t (track etch rate) and V_b (bulk etch rate). As it is known that V_b depends on many factors like structure, chemical composition and preparation of the detector, etching conditions (like chemical nature of etchant, temperature, concentration as well as the stirring conditions) aging, pre-irradiation treatment and storage conditions, environmental conditions during irradiation (ionizing as well as the non ionizing radiation) etc. [12]. Various techniques have been used for the determination of the bulk etch rate [13–17], however we used direct measurement technique discussed by Mehta *et al.* and Shikha *et al.* [18–19]. As the chemical etching technique is widely used to measure the bulk etch rates of LR-115, its surface chemical etching properties should be studied in more details. Among other information, the bulk etch characteristics will be vital in the practical use of this type of SSNTD in radon measurement experiments. The present paper is devoted to determine the bulk etch rates of the LR-115 detectors (Type II, non-strippable, purchased from DOSIRAD, France) under different etching conditions, which are then generalized in the form of an activation energy in the Arrhenius equation.

2. EXPERIMENTAL DETAILS

Pristine LR 115 type-II (cellulose nitrate) was procured from DOSIRAD, France in the form of thin films of active layer thickness of 12 µm on a clear 100 µm polyester base. Several samples of size 3.5 × 3.5 cm were cut from the films. In order to ascertain the effect of temperature and normality on bulk etch rate, two different studies were made. Two sets of the samples were made; each set is having three samples in it for this study so as to reduce the error in the results. The samples of one set were etched in 2.5 N NaOH in a constant temperature water bath at 40°C, 50°C, 60°C and 70°C ± 0.1°C for different time intervals in the steps of 20 minutes (except at 70°C where the etching rate was very high, so 10 minutes time interval step was taken) each without stirring as shown in Figure 1. After each etching interval the samples were washed thoroughly in distilled water and then dried in open air for 30 minutes. The etchant was changed periodically so that concentration of etchant remained the same during the experiment. The thickness measurements for a particular sample were taken before & after etching on a specified area. Etch rate was determined by measuring the foil thickness using a digital micrometer (Mitutoyo, No. 293–821) having least count of 1 µm.

In order to ascertain the effect of normality of the etchant, the samples of second set were etched 1.5 N NaOH, 2.5 N NaOH and 3.5 N NaOH at 60°C ±

0.1°C in a constant temperature water bath for different time intervals in the steps of 20 minutes each without stirring. For the measurement of the thickness the same procedure was used as mentioned above.

Fig. 1 – a) Constant temperature water bath; b) sample etching arrangement in the water bath.

3. RESULTS AND DISCUSSION

The surface chemical etching behavior of LR 115 type-II was studied by measuring the cumulative thickness (total etched-out thickness of the sample, as measured by the digital micrometer before and after etching), removed. The measured values of cumulative thickness removed (in µm) for LR-115 type-II etched in both the conditions *i.e.* different temperatures and different etchant normality. For understanding the effect of temperature on surface chemical etching behavior of LR-115, the test pieces were subjected to etching without stirring in a same etchant 2.5 N NaOH, but at different temperatures 40°C, 50°C, 60°C and 70°C ± 0.1°C. The measured values are given in Table 1 and Table 2.

Table 1

Cumulative Thickness Removed (in µm) in 2.5 N NaOH with time at 40°C, 50°C and 60°C ± 0.1°C

Time (min)	Cumulative thickness removed (µm)		
	40°C	50°C	60°C
20	0.0	0.0	1.0
40	1.0	1.0	2.0
60	1.0	2.0	4.0
80	2.0	3.0	5.0
100	2.0	3.0	7.0

Table 2

Cumulative Thickness Removed (in µm) in 2.5 N NaOH with time at 70°C ± 0.1°C

Time (min)	10	20	30	40	50
Cumulative thickness removed (µm)	2.0	6.0	8.0	9.0	10.0

The data provided in the tables was used to produce Fig. 2 and Fig. 3 that gives the average bulk etch rates for the LR-115 as 1.37 µm/h, 2.14 µm/h, 4.2 µm/h and 12.5 µm/h for 40°C, 50°C, 60°C and 70°C ± 0.1°C respectively. Figure 3 and Fig. 4 show a substantial increase in the average bulk etch rate of the polymer with the increase of temperature.

For understanding the effect of etchant normality on surface chemical etching behavior of LR-115, the test pieces were subjected to etching without stirring in a same etchant, at same temperature but with different etchant normality *i.e.* in 1.5 N NaOH, 2.5 N NaOH and 3.5 N NaOH at 60°C ± 0.1°C. Table 3 shows the values of cumulative thickness removed. The data provided in table was used to produce Figure 5 that gives the average bulk etch rates for the LR-115 as 3.0, 4.2 and 5.2 µm/h for 1.5 N, 2.5 N and 3.5 N NaOH normality respectively. Figure 4 shows some increase in the average bulk etch rate of the polymer with the increase of etchant normality. This is due to the fact that once the etching starts more etchant concentration leads to the increase in etching of the polymer (concentration effect) and hence more average bulk etch rate.

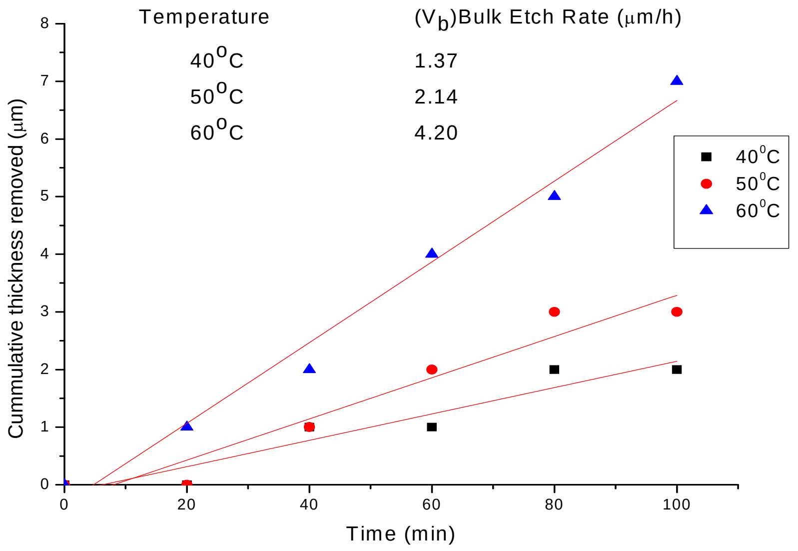

Fig. 2 – Effect of temperature on variation on cumulative thickness removed of LR-115 SSNTD at 40°C, 50°C and 60°C ± 0.1°C.

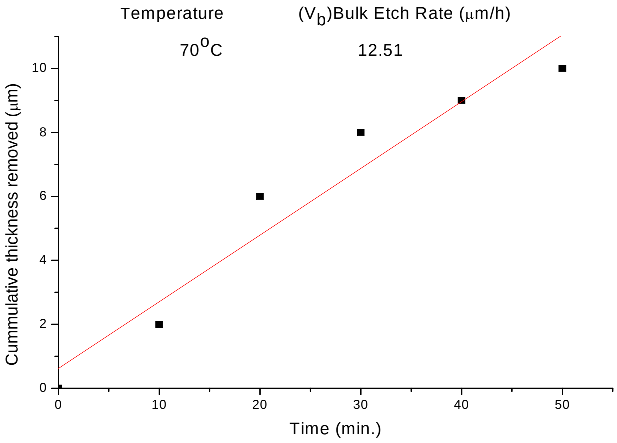

Fig. 3 – Effect of temperature on variation on cumulative thickness removed of LR-115 SSNTD at 70°C ± 0.1°C.

Table 3

Effect of Normality of NaOH on Cumulative Thickness removed with time at 60°C ± 0.1°C

Time (min)	Cumulative thickness removed (μm)		
	1.5 N NaOH	2.5 N NaOH	3.5 N NaOH
20	1.0	1.0	1.0
40	2.0	2.0	3.0
60	3.0	4.0	6.0
80	4.0	5.0	7.0
100	5.0	7.0	8.0

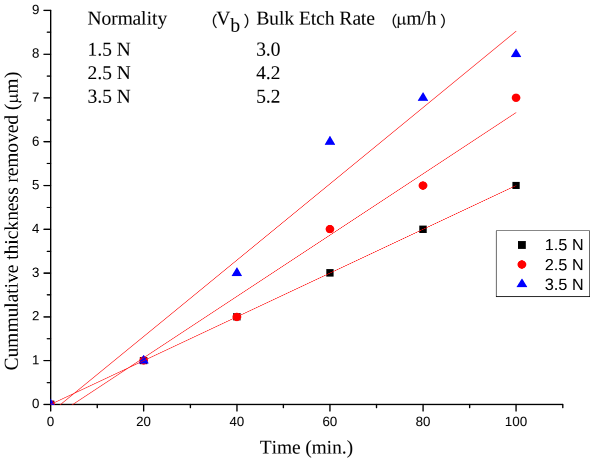

Fig. 4 – Effect of normality of NaOH on variation of cumulative thickness removed at 60°C ± 0.1°C.

Table 4 gives the values of average bulk etching rate of LR-115 type-II SSNTD at different etching temperature for 2.5 N aqueous solution of NaOH.

Table 4

Average bulk-etch rate of LR-115 type II SSNTDs at various etch temperatures for 2.5 N NaOH, without stirring

Etching Temperature	40°C	50°C	60°C	70°C
Average bulk-etch rate (V_b, μmh^{-1})	1.37	2.14	4.20	12.51

The activation energy (E_b) values of bulk etching for the LR-115 type II SSNTD was calculated from the slope of the line plot, logV_b *vs* 1000/T (Fig. 5).

$$V_b = A \exp(-E_b/kT), \quad (1)$$

where A is a constant that depends upon the nature of the material, k is Boltzmann's constant and T is the temperature in K. The E_b is found to be 0.668 eV for these samples.

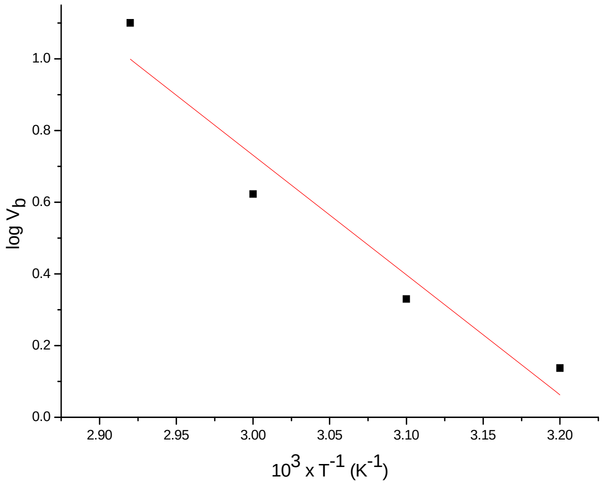

Fig. 5 – Graph of log V_b *versus* $10^3 \times T^{-1}$, with T in K for LR-115 type-II SSNTD.

4. CONCLUSIONS

The surface chemical behaviour of LR-115 SSNTD was studied by etching process. The thickness of the polymeric sample at different temperatures and normalities was measured for finding the bulk etch rates. The result indicated an increase in etch rate with temperature and normality. The activation energy (E_b) values of bulk etching for the LR-115 type II SSNTD was calculated and was found to be 0.668 eV. This value of activation energy is in close agreement with the value of activation energy of 0.676 eV as reported by Tse *et al.* for the etching of colorless LR-115 in NaOH without stirring [16].

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