Available online at www.sciencedirect.com
SCIENCE DIRECT®
Radiation Measurements 38 (2004) 1–3
Radiation Measurements
www.elsevier.com/locate/radmeas

Non-destructive measurement of active-layer thickness of LR 115 SSNTD

F.M.F. Ng, C.W.Y. Yip, J.P.Y. Ho, D. Nikezic[1], K.N. Yu*

Department of Physics and Materials Science, City University of Hong Kong, Tat Chee Avenue, Kowloon Tong, Kowloon, Hong Kong

Received 28 July 2003; accepted 15 September 2003

Abstract

LR 115 is a solid-state nuclear track detector (SSNTD) based on cellulose nitrate and has been commonly used for measurements of concentrations of radon gas and/or radon progeny. These measurements depend critically on the removed thickness of the active layer during etching. However, the thickness of removed layer calculated using the etching period does not necessarily provide a sufficiently accurate measure of the thickness. For example, the bulk etch rate depends on the strength of stirring during etching. We propose here to measure the thickness of the active layer by using Fourier Transform Infrared (FTIR) spectroscopy, with the wave number at 1598 cm^{-1} corresponding to the O–NO_2 bond. We have found an exponential decay relationship between the infrared transmittance at the wave number at 1598 cm^{-1} and the thickness of the active layer for LR 115 detector. This provides a fast and non-destructive method to measure the thickness of removed layer from etching of LR 115 detector.

Keywords: Solid-state nuclear track detector (SSNTD); LR 115; Active layer; FTIR

1. Introduction

It has been well established that inhalation of short-lived progeny of radon (^{222}Rn) can cause lung cancers. This has attracted much attention to the exploration of long-term passive integration measurement methodologies regarding short-lived radon progeny. There are a few different approaches to this problem, all of them being based on the usage of solid-state nuclear track detectors (SSNTDs). Alpha particles emitted by ^{222}Rn and its progeny hit the detector and leave latent tracks in it, which are made visible by chemical or electrochemical etching. One of the directions nowadays is to exploit alpha spectroscopy (Mozzo et al., 1996), while another one is to explore particular etching conditions in such a way that only tracks with specified characteristics or in particular energy windows are seen on the detector (Baixeras et al., 1999). All these methods rely on an accurately defined thickness of etched layer of the detector.

One of the most commonly used SSNTDs is the LR 115 detector (see e.g., Nikolaev and Ilic, 1999), which is based on cellulose nitrate. Yip et al. (2003a) showed that the bulk etch rate of this detector could not be controlled by temperature and etchant concentration only, and was also affected by the amount of stirring. Therefore, actual monitoring of the active-layer thickness is necessary when using the LR 115 detector. Surface profilometry has been proposed by Nikezic and Janicijevic (2002) to measure the active-layer thickness of the LR 115 SSNTD, but this is a destructive method, so the method can only be used after etching has been completed (referred to as a posteriori method). An a priori method was subsequently proposed by Yip et al. (2003b) to measure the thickness of the active layer of LR 115 SSNTDs before the completion of etching, which was based on the absorption of fluorescence X-ray photons by the active layer. However, there is a risk that X-ray radiation affects the track and bulk etching velocities. For example, Clark and Stephenson (1982) and

* Corresponding author. Tel.: +852-2788-7812; fax: +852-2788-2830.
E-mail address: peter.yu@cityu.edu.hk (K.N. Yu).
[1] On leave from University of Kragujevac, Serbia and Monte Negro.

1350-4487/$ - see front matter © 2003 Elsevier Ltd. All rights reserved.
doi:10.1016/j.radmeas.2003.09.001

Fowler et al. (1985) have shown X-ray degradation of cellulose nitrates.

In the present work, the absorption of infrared radiation by the active layer will be explored as a means for determination of its thickness. Infrared spectroscopy is a non-destructive method, which has been used by Barillon et al. (2002) to quantify the loss of nitrate functions in the LR 115 SSNTD under ion irradiation.

2. Experiment

The LR 115 detectors used in the present study were purchased from DOSIRAD, France (LR 115 film, Type 2, non-strippable). The detectors consist of an active layer of red cellulose nitrate on a 100 μm clear polyester base substrate. A total of 11 pieces of LR 115 detectors were employed for our experiments, each with a size of about 1.5×2 cm^2.

To establish the correlation between the infrared transmittance and the thickness of the active layer of the LR 115 detector, the active-layer thickness should be accurately determined. The procedures for measuring the active-layer thickness followed those devised by Nikezic and Janicijevic (2002). Before etching, a small portion of the red cellulose nitrate layer was first removed by a razor to expose the colorless polyester base. The detectors were then etched separately in a 2.5 N aqueous solution of NaOH maintained at 60°C by a water bath, which is the most frequently used etching condition for the LR 115 detector. The temperature was kept constant with an accuracy of ±1°C. The detectors were etched using a magnetic stirrer (Model No: SP72220-26, Barnstead/Thermolyne, Iowa, USA) for more uniform etching (Yip et al., 2003a).

At different etching periods, the detectors were removed from the etchant and immediately rinsed with distilled water. After drying, the portions of the detectors with the active layer were scanned using a Perkin-Elmer Fourier Transform Infrared (FTIR) spectroscopy system (Model 16 PC FT-IR) for 10 cycles. The wave number range employed was between 1700 and 1100 cm^{-1}, with a resolution of 4 cm^{-1}. The scanned diameter was 9 mm so the scanned area was 0.64 cm^2. A spectrum showing the variation of the infrared transmittance (%) with the wave number (cm^{-1}) would be generated by the FTIR system after scanning.

After the FTIR measurements, the thickness of the active layer was revealed by surface profilometry measurements. A surface profilometry system called Form Talysurf PGI (Taylor Hobson, Leicester, England) was employed to measure the active-layer thickness. The measuring system is based on a laser interferometric transducer. A computer-controlled stylus passes slowly across a surface of interest during measurements, while the data are processed by the computer to generate an output graph showing the profile of the scanned surface. The mean value and the standard deviation for the active-layer thickness were obtained through measurements for five different positions of the cliff.

3. Results and discussion

As mentioned before, the wave number range employed was between 1700 and 1100 cm^{-1}. The wave number most sensitive to the thickness of the active layer was identified as 1598 cm^{-1}. The infrared absorption peak was the largest at this wave number for a detector before etching, while no absorption peak was found at this wave number for the polyester base alone. This wave number was similar to the wave number 1600 cm^{-1} identified by Barillon et al. (2002) as being contributed by the O–NO_2 bond in the cellulose nitrate in the LR 115 SSNTDs. In all subsequent discussions, the infrared transmittance refers to the value measured at the wave number 1598 cm^{-1}.

The active-layer thickness for LR 115 measured by surface profilometry and the corresponding infrared transmittance are shown in Fig. 1. An anti-correlation between the two variables is apparent. By fitting the exponential decay relationship $y = A\exp(-Bx)$ to the experimental data, where y (μm) is the thickness of the active layer measured by surface profilometry, and x (%) is the infrared transmittance, we have $A = (19.7 \pm 0.3)$ and $B = (0.111 \pm 0.003)$, with $R^2 = 0.9958$. This relationship with a large value of R^2 and the narrow 95% confidence band demonstrate that the active-layer thickness for LR 115 can be satisfactorily reflected by the infrared transmittance.

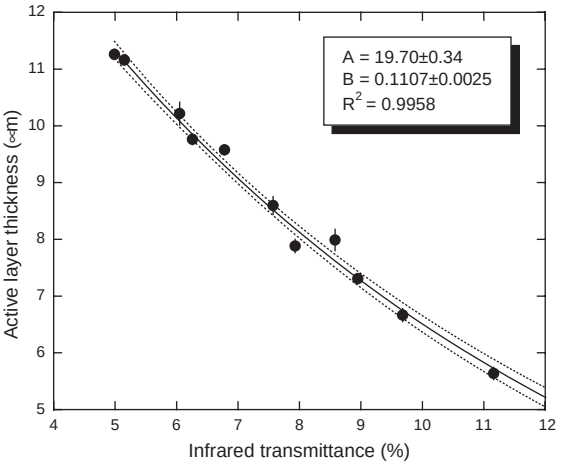

Fig. 1. Relationship between the active-layer thickness for the LR 115 SSNTD measured by surface profilometry and the corresponding FTIR transmittance at the wave number 1598 cm^{-1}. Some error bars are smaller than the symbols. The solid line is the best-fit exponential decay curve represented by $y = A\exp(-Bx)$, with the parameters A and B shown. The dotted lines represent the 95% confidence bands.

Moreover, the fit is observed to be valid within the entire range of the present data set (active-layer thickness from 5.6 to 11.3 μm). For the standard etching conditions that are usually applied to LR 115 SSNTDs, namely, the etchant of the 2.5 N aqueous solution of NaOH kept at 60°C, the bulk etch rate under magnetic stirring has been found to be 6.65 ± 0.34 μm h^{-1}, while that under no stirring to be 3.61 ± 0.14 μm h^{-1} (Yip et al., 2003a). By adopting the nominal initial thickness of the active layer in the LR 115 detector of 12 μm, although some variations have been found (Yip et al., 2003a), the above relationship is valid for etching durations up to about 1 and 1.8 h under magnetic stirring and no stirring, respectively. In this way, we have successfully established a fast and non-destructive a priori technique based on FTIR to determine the active-layer thickness of LR 115 SSNTDs.

We have also attempted to extend the relationship to smaller active-layer thickness. However, for active-layer thickness smaller than about 5 μm, the surface profilometry data became significantly more scattered. This agrees with our previous finding of increasing inhomogeneity in the active layer with the etching duration (Yip et al., 2003c). For long-term measurements of short-lived radon progeny which rely on accurately defined thickness of etched layer of the detector, excessive inhomogeneity might cause a problem. From the present experimental results, active layers with a thickness larger than 5.6 μm will be suitable if a well-defined thickness is required.

Acknowledgements

The present research is supported by the CERG Grants CityU1081/01P and CityU1206/02P from the Research Grant Council of Hong Kong.

References

Baixeras, C., Amgarou, K., Font, L.L., Domingo, C., Fernandez, F., 1999. Measurement of the ^{214}Po concentration in air using Markrofol-DE detectors. Radiat. Meas. 31, 313–318.

Barillon, R., Fromm, M., Katz, R., Chambaudet, A., 2002. Chemical bonds broken in latent tracks of light ions in plastic track detectors. Radiat. Prot. Dosim. 99, 359–362.

Clark, D.T., Stephenson, P.J., 1982. Electron-spectroscopy for chemical applications (ESCA) applied to polymers — an ESCA study of the surface-chemistry of cellulose nitrates and double based propellants, with particular reference to their degradation in UV-light. Polym. Degradat. Stabil. 4, 185–193.

Fowler, A.H.K., Munro, H.S., Clark, D.T., 1985. ESCA studies of the thermal and X-ray induced degradation of cellulose nitrates. Polym. Degradat. Stabil. 11, 287–296.

Mozzo, P., Trotti, F., Temporin, A., Lanciai, M., Predicatori, F., Righetti, F., Tacconi, A., 1996. α-spectroscopy on CR-39 track detectors for the dosimetry of radon daughters. Environ. Int. 22 (Suppl. 1), S595–S600.

Nikezic, D., Janicijevic, A., 2002. Bulk etching rate of LR-115 detectors. Appl. Radiat. Isot. 57, 275–278.

Nikolaev, V.A., Ilic, R., 1999. Etched track radiometers in radon measurements: a review. Radiat. Meas. 30, 1–13.

Yip, C.W.Y., Ho, J.P.Y., Koo, V.S.Y., Nikezic, D., Yu, K.N., 2003a. Effects of stirring on the bulk etch rate of LR 115 detector. Radiat. Meas. 37, 197–200.

Yip, C.W.Y., Ho, J.P.Y., Nikezic, D., Yu, K.N., 2003b. A fast method to measure the thickness of removed layer from etching of LR-115 detector based on EDXRF. Radiat. Meas. 36, 161–164.

Yip, C.W.Y., Ho, J.P.Y., Nikezic, D., Yu, K.N., 2003c. Study of inhomogeneity in thickness of LR 115 detector with SEM and Form Talysurf. Radiat. Meas. 36, 245–248.