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Measuring depths of sub-micron tracks in a CR-39 detector from replicas using Atomic Force Microscopy

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Abstract

One of the challenging tasks in the application of solid-state nuclear track detectors (SSNTDs) is the measurement of the depth of the tracks, in particular, the shallow ones resulting from short etching periods. In the present work, a method is proposed to prepare replicas of tracks from α particles in the CR-39 SSNTDs and to measure their heights using atomic force microscopy (AFM). After irradiation, the detectors were etched in a 6.25 N aqueous solution of NaOH maintained at 70 °C. The etched detectors were immersed into a beaker of the replicating fluid, which was placed in a water bath under ultrasonic vibration and maintained at room temperature to facilitate the filling of the etched tracks with the replicating fluid. As an example of application, these results have been used to derive a V function for the CR-39 detectors used in the present study (for the specified etching conditions).

Keywords: CR-39; Track length; Replica; Atomic force microscopy

1. Introduction

Solid-state nuclear track detectors (SSNTDs) have found applications in different branches of science (see e.g. Nikezic and Yu, 2004). One of the challenging tasks in the application of SSNTDs is the accurate measurement of the depth of the tracks, e.g., those from α particles, in particular, the shallow ones with sub-micron lengths resulting from short etching periods.

Indirect measurements of α-track depths are usually performed by optical methods. While measurements of track-opening diameters are relatively straightforward, some researchers might need or prefer direct measurements of track lengths. One approach involves the breaking of SSNTDs to reveal the lateral images of the tracks for direct measurements (Dörschel et al., 1997) and another involves the use of confocal microscopy (Vaginay et al., 2001). For relatively long α tracks (e.g., more than 10 μm), i.e., those resulting from relatively long etching time, surface profilometry has been proposed to measure the heights of the replicas of the α tracks (Yu et al., 2004). As mentioned before, however, depths of shallow tracks (e.g., in the sub-micron range) resulting from short etching periods are sometimes needed.

In the present work, we propose a method based on atomic force microscopy (AFM) to determine the lengths of tracks in CR-39 detectors through measurements of their replicas. The replicas are required because of the geometry of the probe of the AFM which may prevent the probe from reaching the bottom of the tracks (Nikezic et al., 2002). It is also emphasized that the track profile is not our focus in the present study.

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2. Methodology

The CR-39 detectors used in the present study were purchased from Page Mouldings (Pershore) Limited (Worcestershire, England). The original dimensions of a sheet of the detector were 30 cm × 47 cm × 0.1 cm (thickness). The detectors for our studies were cut to a size of $1 \times 1\,\text{cm}^2$. The CR-39 detectors were irradiated with α particles with energies from 1 to 4.5 MeV, with steps of 0.5 MeV under normal incidence through a collimator. The α source employed in the present study was a planar ^{241}Am source (main α energy = 5.49 MeV under vacuum). Normal air was used as the energy absorber to control the final α energies incident on the detector. A relationship between the α energy and the air distance travelled by an α particle (with an initial energy of 5.49 MeV from ^{241}Am) was therefore needed. This relationship was obtained by measuring the energies for α particles passing different distances through normal air using α spectroscopy systems (ORTEC Model 5030) with passivated implanted planar silicon (PIPS) detectors of areas of $300\,\text{mm}^2$.

After irradiation, the detectors were etched in a 6.25 N aqueous solution of NaOH maintained at 70 °C by a water bath for 15 min, so that the track lengths were in the sub-micron range. The detectors were then taken out from the etchant, rinsed with distilled water and dried in air. A plastic mould in the form of a cylindrical cup with a detachable bottom was used to prepare the resin replicas using Buehler fast cure epoxy (41 Waukegan Rd., Lake Bluff, IL 60044, USA). The inside surface of the mould was first coated with the Buehler release agent (No. 20-8185-002). A piece of etched detector (with the α tracks) was then placed inside the mould on the bottom, with the side containing the tracks facing upwards. The replicating fluid was prepared with Buehler Epo-Kwick resin (No. 20-8136-128) and Buehler Epo-Kwick hardener (No. 20-8138-032) with a mass ratio of 5:1, and was poured into the mould. After drying for more than 10 h, the mould was detached. The CR-39 detector was then removed from the resin after sawing along some edges of the CR-39 detector, leaving behind the replicas of the tracks protruding from the resin surface.

AFM was employed to capture the surface topography and the heights of the track replicas. The AFM used in the present project was the autoprobe CP model from Park Scientific Instruments (1171 Borregas Avenu, Sunnyavale, CA 94089). The probe of the AFM employed was an Ultralever, with an opening angle of 10° and a length of 4 μm. Contact mode operation was used where a high-resolution image was expected. A constant force of 13.2 nN was applied on the tip and the scan rate was 1 Hz. The surfaces of the detectors were imaged directly in air and room temperature.

During measurements, the Ultralever scanned the studied surface many times, with a 256 × 256 and 512 × 512 pixel resolution for a scanning area of $5 \times 5\,\mu\text{m}^2$ and $10 \times 10\,\mu\text{m}^2$, respectively. From the lateral view containing the track replicas recorded by the AFM, the replica heights can be obtained for the corresponding α energy.

3. Results and discussion

Fig. 1 shows a three-dimensional SEM image (viewing directly from above) of replicas of tracks resulted from normally incident 1 MeV α particles and 15-min etching. For the replicas to truly reflect the dimensions of the tracks, the replicating fluid should fill the tracks completely. From Fig. 1, we can see that the surfaces of the replicas are smooth, which is a necessary (although not sufficient) condition to that the replicating fluid fills the tracks completely. A more direct evidence has been provided by the optical microscopic image showing the cross-sections of longer tracks with the dried replicating fluid inside (Yu et al., 2004), which proves that the replicating fluid fills the tracks completely.

Fig. 2(a) shows a three-dimensional image of the replicas recorded by AFM, while Fig. 2(b) shows the corresponding two-dimensional image from the lateral side. The heights of the replicas can be conveniently read from Fig. 2(b). Again, we can see from Fig. 2 that the heights of the replicas are very uniform. The mean heights of the replicas of tracks from α particles with different energies are shown in Table 1 as well as in Fig. 3.

The above procedures have demonstrated the convenience of measuring the track lengths by AFM measurements using replicas of the tracks. As protruding objects are being measured, the track lengths will not be distorted due to artefacts produced by the geometry of the probe of the AFM equipment (Nikezic et al., 2002). It is emphasized that the track profile is not our focus in the present study.

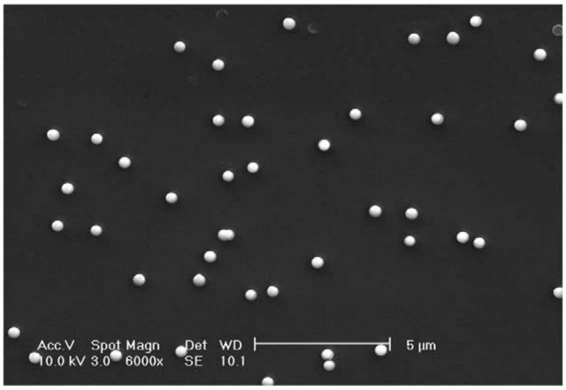

Fig. 1. Three-dimensional SEM image (viewing directly from above) of replicas of tracks resulting from normally incident 1 MeV α particles and 15-min etching.

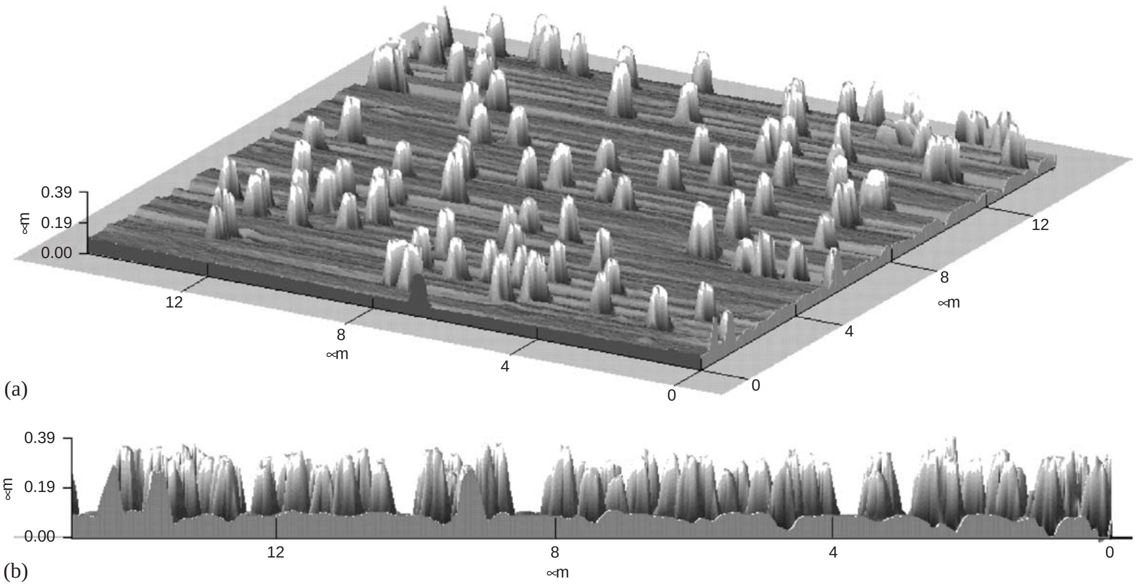

Fig. 2. (a) Three-dimensional image of the replicas of tracks resulting from normally incident 4.5 MeV α particles and 15-min etching recorded by the AFM. (b) Lateral view of the replicas shown in (a). The average height of the replicas is 0.25 ± 0.03 μm.

Table 1
The mean heights of the replicas of tracks from α particles with different energies

Energy (MeV)	Mean height (μm)
1.00 ± 0.12	0.68 ± 0.08
1.50 ± 0.10	0.63 ± 0.08
2.00 ± 0.09	0.62 ± 0.09
2.50 ± 0.08	0.39 ± 0.04
3.00 ± 0.07	0.41 ± 0.13
3.50 ± 0.06	0.28 ± 0.05
4.00 ± 0.06	0.27 ± 0.04
4.50 ± 0.05	0.25 ± 0.03

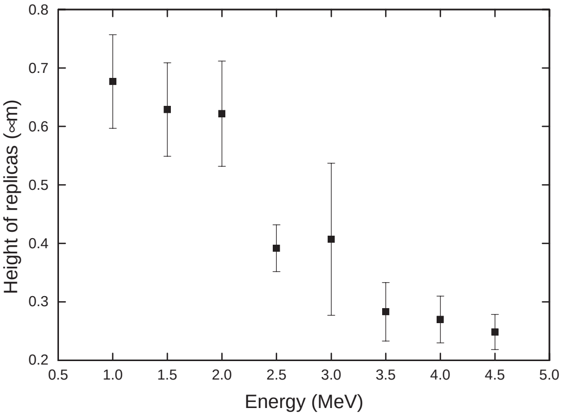

Fig. 3. The mean heights of replicas of tracks from α particles with different energies.

4. An example of application—determination of *V* function

In this section, we will show one example of application of the AFM measurements of replicas in the determination of the *V* function for the CR-39 detectors used in the present study (for the specified etching conditions). Much research has been devoted to understanding the mechanisms of track growth in SSNTDs. The most widely accepted track growth model involves two etch rates, namely, the track etch rate V_t (i.e., along a track in the SSNTD) and the bulk etch rate V_b (i.e., the undamaged areas of the SSNTD).

From the data shown in Table 1, it is possible to determine V_t fairly accurately by assuming that V_t is relatively constant in a short period of etching. The track length L represented by the replica height is given as

$$L = V_t t - V_b t, \tag{1}$$

where t is the etching time. Therefore,

$$V = \frac{V_t}{V_b} = 1 + \frac{L}{h}, \tag{2}$$

where h is the layer removed during the etching time t. Here we have made use of a bulk etch rate of 1.2 μm h^{-1} determined previously for the CR-39 detectors used in the

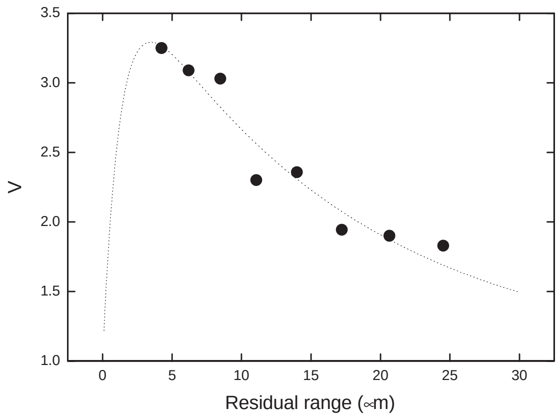

Fig. 4. V as a function of the residual range of the α particles with different energies. Solid circles: experimental points; Dotted line: best-fit V function.

present study (Ho et al., 2003). Since the etching time was 15 min, $h = 0.3\,\mu\text{m}$.

To obtain the V function, we use a parametric function in the form

$$V = 1 + e^{-a_1 R+b} - e^{-a_2 R+b} \tag{3}$$

with three parameters, viz. a_1, a_2 and b, where R represents the residual ranges of the α particles in the CR-39. The residual ranges were calculated by means of the SRIM2003 code (http://www.srim.org/). The best fit of Eq. (3) to the V values calculated with Eq. (2) for each of the α-particle energy used in the experiment gives the parameters as $a_1 = 0.06082$, $a_2 = 0.8055$ and $b = 1.119$. This best function together with the experimental data are shown in Fig. 4.

Brun et al. (1999) showed (in their Fig. 4) that, with an etchant of 6 N NaOH at 70 °C (which is closest to our etchant of 6.25 N NaOH at 70 °C), the V_t values were about 5 and $2.5\,\mu\text{m/h}^{-1}$ at the residual ranges of 4 and 30 μm, respectively. To calculate the corresponding V values, we need the V_b value for their detectors and their etching conditions. If we adopt $V_b \propto C^{3/2}$ (Somogyi and Hunyadi, 1979), we can estimate their V_b value to be about $1.7\,\mu\text{m/h}^{-1}$, so $V \approx 3$ and 1.5 at the residual ranges of 4 and 30 μm, respectively. We can therefore see that the V values obtained in the present paper agree very well with the results presented by Brun et al. (1999), despite the different manufacturers of the CR-39 detectors.

Acknowledgements

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References

Brun, C., Fromm, M., Jouffroy, M., Meyer, P., Groetz, J.E., Abel, F., Chambaudet, A., Dörschel, B., Hermsdorf, D., Bretschneider, R., Kadner, K., Kunhe, H., 1999. Intercomparative study of the detection characteristics of the CR-39 SSNTD for light ions: present status of the Besancon-Dresden approaches. Radiat. Meas. 31, 89–98.

Dörschel, B., Fülle, D., Hartmann, H., Hermsdorf, D., Kadner, K., Radlach, Ch., 1997. Measurement of track parameters and etch rates in proton-irradiated CR-39 detectors and simulation of neutron dosemeter responses. Radiat. Prot. Dosim. 69, 267–274.

Ho, J.P.Y., Yip, C.W.Y., Nikezic, D., Yu, K.N., 2003. Effects of stirring on the bulk etch rate of CR-39 detector. Radiat. Meas. 36, 141–143.

Nikezic, D., Yu, K.N., 2004. Formation and Growth of Tracks in Nuclear Track Materials. Mater. Sci. Eng. R 46, 51–123.

Nikezic, D., Ho, J.P.Y., Yip, C.W.Y., Koo, V.S.Y., Yu, K.N., 2002. Feasibility and limitation of track studies using Atomic Force Microscopy. Nucl. Instrum. Methods Phys. Res. J. (Section B) 197, 293–300.

Somogyi, G., Hunyadi, I., 1979. Etching properties of the CR-39 polymeric nuclear track detectors. 1980. Proceedings of the 10th Conference on SSNTD, Lyon, 2–6 July, 1979, 443–452.

Vaginay, F., Fromm, M., Pusset, D., Meesen, G., Chambaudet, A., Poffijn, A., 2001. 3D confocal microscopy track analysis: a promising tool for determining CR-39 response function. Radiat. Meas. 34, 123–127.

Yu, K.N., Ng, F.M.F., Ho, J.P.Y., Yip, C.W.Y., Nikezic, D., 2004. Measurement of parameters of tracks in CR-39 detector from replicas. Radiat. Prot. Dosim. 111, 93–96.