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Determination of alpha-particle track depths in CR-39 detector from their cross-sections and replica heights

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Abstract

A challenging task in the application of solid-state nuclear track detectors (SSNTDs) is the measurement of depths of the tracks. One approach involves breaking and polishing the side of SSNTDs to reveal the cross-sections of the tracks for direct measurements. Recently, surface profilometry was used to measure the heights of the replicas of alpha-particle tracks to give the track depths. In the present work, systematic comparisons among the track depths for alpha-particles with normal incidence and different incident energies were made for these two methods. After irradiation, the detectors were etched in a 6.25 N aqueous solution of NaOH at 70 °C. Both long etching time of 15 h (to produce spherical-phase tracks) and short etching time from 1 to 8 h (to produce sharp-phase tracks) were used. Good agreement was achieved between the two methods for spherical-phase tracks but not for sharp-phase tracks. It has been found that the surface profilometry method only works for replicas for spherical-phase tracks. Replicas for sharp-phase tracks are easier to collapse or deform, so the surface profilometry method may not give correct results.

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Keywords: CR-39; SSNTDs; Track depths; Replicas; Track etch velocity

1. Introduction

Solid-state nuclear track detectors (SSNTDs) have found applications in different branches of science. A recent review on SSNTDs can be found in [1]. Many researches have 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. in undamaged areas of the SSNTD). All proposed methods for track growth should be tested with experimental data including, e.g. the track-opening diameters and the track lengths.

These alpha track parameters are usually determined through measurements of track-opening diameters. While measurements of track-opening diameters are relatively straightforward, direct measurements of track lengths are relatively difficult. One approach involves breaking the SSNTDs from the side to reveal the lateral images or cross-sections of the tracks for measurements [2] and another involves the use of confocal microscopy [3].

Recently, a method based on contact stylus profilometry was proposed to determine the lengths of tracks in CR-39 detectors through measurements of their replica heights [4]. Replicas were required because the geometry of the stylus of the profilometer may prevent the stylus from reaching the bottom of the tracks. For the sake of demonstrating the applicability of the method, only normally incident 4 MeV alpha-particles were used, and only spherical-phase tracks (etched in a 6.25 N aqueous solution of NaOH at 70 °C for 15 h) were used.

In the present work, the track lengths obtained from this surface profilometry method (hereafter referred to as the replica method) will be compared with those obtained from measurements of lateral images of the tracks (hereafter

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referred to as the cross-section method). Systematic comparisons will be made for incident energies from 1 to 4.5 MeV with steps of 0.5 MeV. Both sharp-phase and spherical phase tracks will be studied. The results will provide more confidence in using the replica method and will also be useful for understanding the limitations of the method.

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 are $30\ cm \times 47\ cm \times 0.1\ cm$ (thickness). The detectors for our studies were cut to a size of $1.5 \times 1.5\ cm^2$. The CR-39 detectors were irradiated with alpha-particles with energies from 1 to 4.5 MeV, with steps of 0.5 MeV, under normal incidence through an acrylic resin collimator with a hole diameter of 1 mm. The alpha source employed in the present study was a planar ^{241}Am source (main alpha energy = 5.49 MeV under vacuum). Normal air was used as the energy absorber to control the final alpha energies incident on the detector. A relationship between the alpha energy and the air distance traveled by an alpha-particle (with initial energy of 5.49 MeV from ^{241}Am) was therefore needed. This relationship was obtained by measuring the energies for alpha-particles passing different distances through normal air using α spectroscopy systems (ORTEC Model 5030) with passivated implanted planar silicon (PIPS) detectors of areas of $300\ mm^2$. After irradiation, the CR-39 detectors were etched in a 6.25 N aqueous solution of NaOH maintained at 70 °C resulting in a bulk etch rate of V_b of about 1.2 μm/h [5]. Different etching durations were used to produce sharp-phase and spherical-phase tracks (see Table 1). After chemical etching, the detectors were removed from the etchant, rinsed with distilled water and dried in air.

2.1. Track depth measurement by cross-section method

For direct measurements of track depths, the etched detectors with alpha tracks were first polished from their sides. The edges were positioned under the microscope so that the cross-sections of the etch pits could be visualized under the optical microscope [1]. Images were recorded in the JPEG format by a digital camera (Olympus DP 11) attached to the microscope. The ImageJ (Image Processing and Analysis in Java) software (version 1.29x) (http://rsb.info.nih.gov/ij/) was employed to analyze the track depths. ImageJ is a powerful tool which can perform most image processing or analysis tasks, including measurements of spatial dimensions. A spatial calibration, through an image of a ruler, is required to provide conversions to real world dimensions.

Table 1
Etching duration (h) for different alpha energies

Alpha energy (MeV)	Sharp-phase tracks	Spherical-phase tracks
1.0	1	15
1.5	1	15
2.0	2	15
2.5	3	15
3.0	4	15
3.5	5	15
4.0	6	15
4.5	8	15

2.2. Replica height measurement by replica method

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) [4]. The inside surface of the mould was first coated with the Buehler Release Agent (No. 20-8185-002). Etched detectors (with the alpha tracks) were placed inside the mould with the irradiated side 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 the mass ratio of 5:1, and was poured into the mould. After drying, the CR-39 detector was removed from the resin.

The replica heights were then measured by the Form Talysurf PGI Profilometer (Taylor Hobson, Leicester, England), which is a contact stylus instrument based on a phase grating interferometric (PGI) transducer. During measurements, a computer-controlled stylus scanned slowly across a surface of the specimen. The movement of the stylus was converted into an electrical signal by the computer and the profile of the scanned surface was generated. The scanning was set to ensure that the probe passed across any one track at least 8 times.

3. Results and discussion

Fig. 1(a) and (b) shows the lateral image of normally-incident 4 MeV alpha-particle tracks after etching in 6.25 N NaOH for 6 h and 15 h, respectively, observed under an optical microscope. Clearly, Fig. 1(a) shows sharp-phase tracks while Fig. 1(b) shows a spherical-phase track.

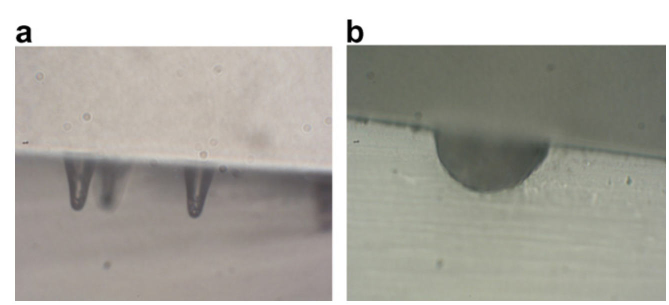

Fig. 1. Lateral images of normally-incident 4 MeV alpha-particle tracks after etching for (a) 6 h and (b) 15 h, respectively, observed under an optical microscope.

The length of the track shown in Fig. 1(b) was obtained from ImageJ as 14.287 μm. Fig. 2(a) shows a two-dimensional image of the replicas of the tracks produced under the same conditions as recorded by Form TalySurf PGI Profilometer. The image with the contours gives a quick estimate of the replica heights. The equipment can also generate the lateral view of the replicas as shown in Fig. 2(b). Here, the replica heights are clearly given as 14.675 μm, which agrees very well with the result obtained from the cross-section method.

The track lengths for different incident alpha energies obtained by both the cross-section and replica methods are shown in Fig. 3 and Table 2. Two very interesting observations have been made from these data. First, the track lengths for different incident alpha energies for etching duration of 15 h obtained by the cross-section and replica methods are almost identical. This confirms that for these spherical-phase alpha-particle tracks, the replica method is both convenient and accurate in providing the lengths of alpha-particle tracks. Second, the track lengths for sharp-phase tracks given by the replica method are much shorter than those given by the cross-section method. Moreover, the replica method did not always give measurable track lengths and did not seem to give a reasonable trend for the change in the track lengths.

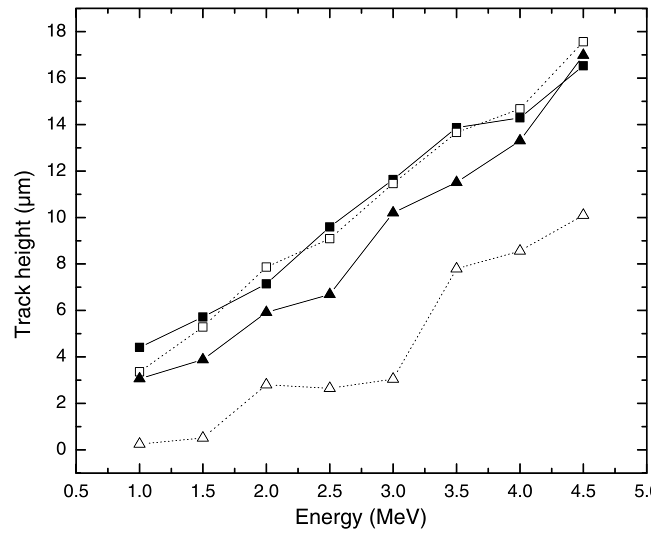

Fig. 3. Track lengths for different incident alpha energies obtained by the cross-section method (solid symbols) and the replica method (open symbols), and for both sharp-phase tracks (triangles) and spherical-phase tracks (squares).

We propose here that contact stylus profilometry can only be applied to replicas for spherical-phase alpha-particle tracks. The replica for conical sharp-phase alpha-parti-

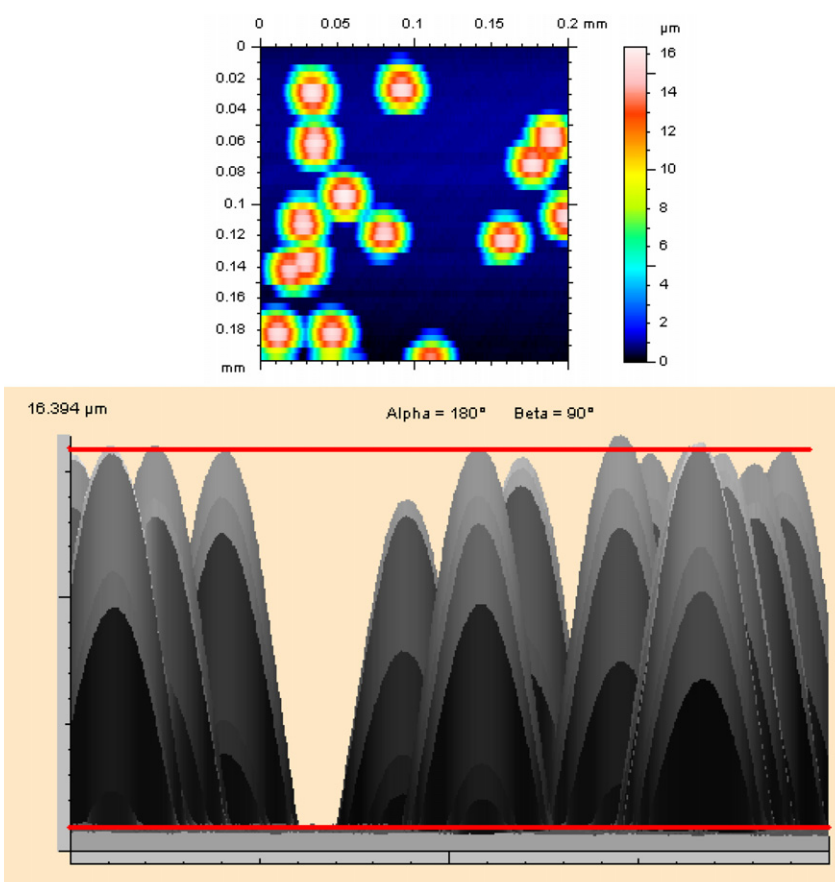

Fig. 2. (a) Two-dimensional image of the replicas recorded by the Form Talysurf PGI surface profilometer. (b) Lateral view of the replicas generated by the Form Talysurf PGI Profilometer. The typical height of the replicas is given by the difference between the two horizontal lines, which is 14.675 μm.

Table 2
Track lengths (μm) for different incident alpha energies obtained by the cross-section and replica methods

Alpha energy (MeV)	Spherical phase		Sharp phase	
	Cross-section method	Replica method	Cross-section method	Replica method
1.0	4.41	3.36	3.06	0.25
1.5	5.72	5.29	3.88	0.51
2.0	7.14	7.87	5.92	2.80
2.5	9.59	9.09	6.69	2.65
3.0	11.63	11.46	10.20	3.05
3.5	13.87	13.66	11.51	7.79
4.0	14.29	14.68	13.30	8.56
4.5	16.53	17.56	16.98	10.10

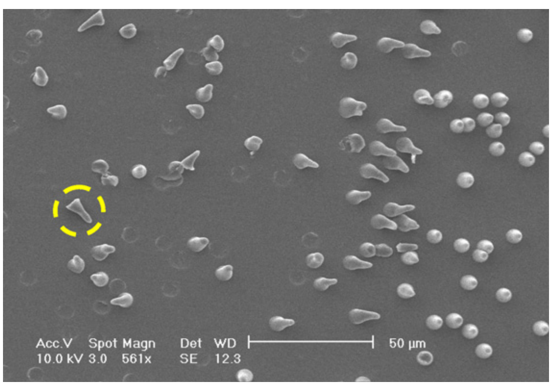

Fig. 4. An SEM micrograph showing the replicas of 3.5 MeV alpha-particle tracks for etching duration of 6 h. The region on the left has been scanned with the Form Talysurf PGI, while the region on the right has not. The circled collapsed replica seems to exhibit its entire length.

cle tracks are less resilient towards the lateral force from the stylus of the profilometer and are thus easier to collapse or deform. Fig. 4 is a very interesting SEM micrograph showing the replicas of 3.5 MeV alpha-particle tracks for etching duration of 5 h, with the region on the left already scanned with the Form Talysurf PGI while the region on the right not scanned. Clearly, most of the replicas in the scanned region have collapsed or deformed, while the replicas in the non-scanned region remain intact. Also shown

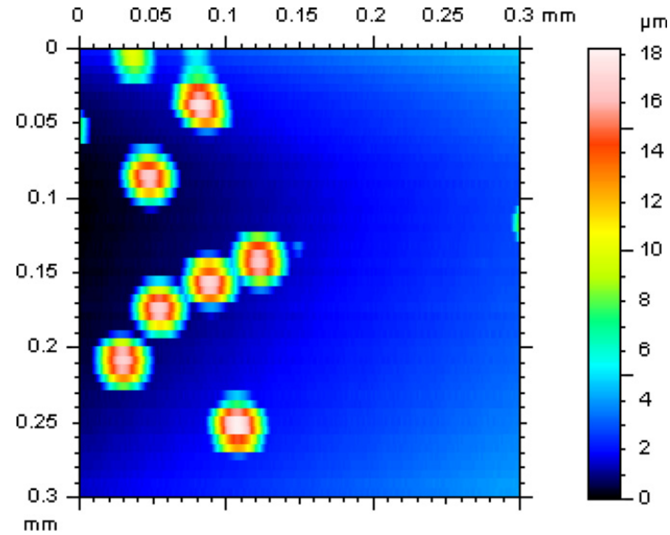

Fig. 6. The image of the square region in Fig. 5 obtained by Form Talysurf PGI.

in the figure is a collapsed replica (circled), which seems to exhibit its entire length. Using the scale on the micrograph, the track length derived from this collapsed replica can be determined as 12 μm. It is interesting that this is very close to the value derived from the cross-section method as 12.24 μm, but is far larger than the average value of 7.79 μm derived from the replica method. These observations give strong support to our conjecture that the replica method does not work for the sharp-phase alpha-particle tracks because they collapse or deform easily during profilometer scans.

In contrast, the contact stylus profilometry works well for spherical-phase alpha-particle tracks. Fig. 5 shows the SEM micrographs of the replicas of 4 MeV alpha-particle tracks for etching duration of 15 h: (a) before scanning with Form Talysurf PGI; (b) after scanning with Form Talysurf PGI. The square region is the area scanned by Form Talysurf PGI, the image of which is shown in Fig. 6. By comparing Fig. 5(a) and (b), we see that the replicas have remained intact, except one which has been completely removed. By referring to Fig. 6, we know that the replica

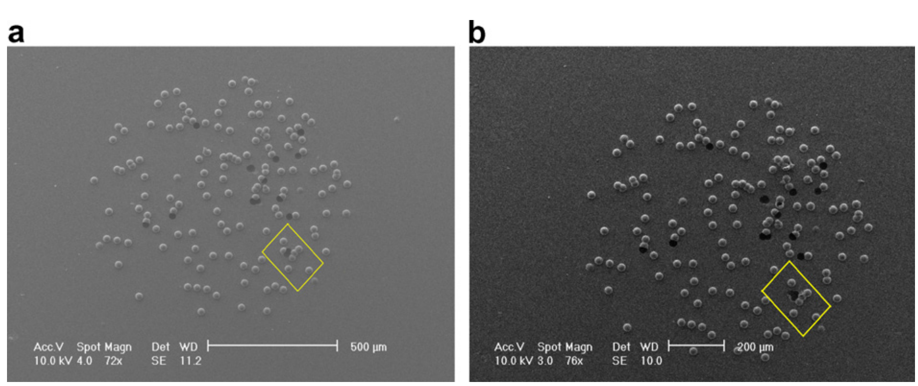

Fig. 5. SEM micrographs showing the replicas of 4 MeV alpha-particle tracks for etching duration of 15 h: (a) before scanning with Form Talysurf PGI; (b) after scanning with Form Talysurf PGI. The square region is the area scanned by Form Talysurf PGI, the image of which is shown in Fig. 6.

has remained intact during the Form Talysurf PGI scan, since its height is the same as all other replicas. The reason for the loss of this replica is not known, but is likely due to sample handling after the profilometer scan.

We therefore come to the conclusion that contact stylus profilometry is a reliable and convenient method in measuring the lengths of spherical-phase tracks, which are in excellent agreement with the corresponding track lengths obtained from the cross-section method. Although the cross-section method is a direct and relatively straightforward method, it has limitations for very short tracks, e.g. from short etching time or small energies. The resolution of the general optical microscope is around 0.3 μm, so the error will become larger than 10% for tracks shorter than 3 μm. In contrast, surface profilometers have much better resolutions. For example, Form Talysurf has a resolution of 0.004 μm. However, surface profilometers cannot be used for sharp-phase tracks as concluded above. For these cases, the non-contact scanning laser profilometers, which use dynamic focusing of the laser beam and focus error signal to determine surface topography, might be useful. Further studies of replicas using such a technique will be carried out in future.

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