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Calibration of CR-39 and ZnP-glass detectors with 350 MeV ^{90}Zr ions

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Abstract. The response of sensitive detectors, CR-39 and ZnP-glass, for 350 MeV ^{90}Zr ions, has been studied. Bulk and track-etch parameters have been evaluated by successive etching. Calibration of the two detectors has been done by correlating the measured track-etch rate (V_T) with the residual range and total energy-loss rate. A linear correlation has been observed in both cases. Track registration threshold values of 4.45 MeV mg^{-1} cm^2 for CR-39 and 12.50 MeV mg^{-1} cm^2 for ZnP-glass have been obtained.

1. Introduction

The versatility of solid-state nuclear track detectors (SSNTDs) [1–8] means that they have become an important probe for various experiments [9, 10], due to the development of new detector materials and the availability of energetic heavy-ion beams. CR-39 and ZnP-glass belong to a group of highly sensitive track detectors among plastics and glasses respectively. The optimisation of an SSNTD for a specific purpose depends mainly on the standardisation of various etching parameters such as bulk-etch rate (V_G) and track-etch rate (V_T) with respect to the rate of energy loss of the track forming heavy ion. The track-etch rate is the best experimentally observable quantity that provides a measure of damage intensity. Theoretically, the range–velocity equations of Bethe [11] and Bohr [12] show a dependence of the energy-loss rate (dE/dx) in any medium on the penetration depths of these heavy ions. Hence, a relationship between V_T and (dE/dx) for any ion, in any medium, provides an understanding of the track-formation mechanism and a calibration curve to identify track-forming particles.

In this paper we present the calibration of the above-mentioned detectors by correlating V_T and dE/dx for ^{90}Zr ions. We have also correlated V_T and the 'residual range'. Maximum etchable track lengths in these detectors have been obtained experimentally and compared with theoretical values [13]. The total times of etching necessary for the detectors have been found by successive etching.

2. Experimental details

2.1. Detector preparation

CR-39 detectors were prepared from commercially available sheets manufactured by Homalite Corporation (Wilmington, DE, USA), by cutting them into 2.0 × 2.0 cm^2 squares.

ZnP-glass detectors were prepared by cutting the thin plates into a size of 2.0 × 2.0 cm^2. The bulk material was a special metaphosphate glass manufactured by Ernst Leitz (AG) (Wetzlar, FRG).

Properties of these detectors are listed in table 1. After

Table 1. Properties of detectors.

Properties	CR-39	ZnP-glass
Composition	$C_{12}H_{18}O_7$	$B_{2.67}O_{68.86}Al_{2.7}Si_{0.35}P_{23.12}Zn_{2.3}$
Chemical name	Allyl diglycol carbonate	Metaphosphate glass
Mol. Wt.	274.0	2079.7
Density (kg m^{-3})	1320	2686
Thickness (μm)	1500	1300
Uniformity	Good	Good
Surface view	Optical grade, no background pits	Optical grade, no background pits
Chemical etchant	NaOH	NaOH
Clarity	Excellent	Excellent
Colour	Colourless	Colourless

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the removal of surface-protecting layers the detectors were washed in soap solution and dried in a vacuum desiccator. The ZnP-glass detectors were polished prior to washing in order to produce a background free and optically plane surface.

2.2. Irradiation

For irradiation, the detectors were mounted on a $5 \times 5\ \mathrm{cm}^2$ glass slide and placed in a special Perspex magazine. All of the samples were exposed to a collimated beam of 350 MeV ^{90}Zr ions at the XO channel of Unilac (GSI, Darmstadt). The detectors were exposed at an incident angle of 45° using an areal dose of $\sim 10^4\ \mathrm{cm}^{-2}$. After irradiation the detectors were preserved in plastic boxes.

2.3. Chemical etching for track development

Latent damage tracks in the detectors were developed by suitable chemical etching. The etchings were carried out in 6N NaOH at 55 °C. The accuracy in the maintenance of temperature was ± 0.5 °C. Prior to etching the detectors were washed with lukewarm soap solution in order to remove all greasy substances from their surfaces. Maximum etchable track lengths were obtained by etching the detectors successively for intervals of 10–15 min. Successive etching helps to determine the rate of increase of track length with respect to time and also enables the time taken to completely etch the tracks to be found. Etching was terminated when rounded track tips were formed. A few test etchings were performed to determine the total etching time for tracks of different lengths in these two detectors. The complete etching time (t_c) depends on etching conditions, the nature of the track-forming ion and its energy. After every etching procedure these detectors were washed in running water for 10–15 min followed by 2–3 washes in distilled water. All etched-track detectors were dried by first pressing them gently between layers of soft filter paper and later in a vacuum desiccator.

2.4. Measurement of track parameters

The length and face diameter of the etched tracks were measured by an optical microscope (Leitz). After every etching the detectors were mounted on a special holder and viewed under the microscope. The detector surface was scanned in both x and y directions in order to identify 10–15 partially etched tracks. For the tracks identified, their diameters were measured as the minor axis of the elliptical face of the track. Projected track lengths were also measured. Similar measurements were carried out for all identified tracks after every etching. The accuracy of measurement was 0.45 μm at ×50 and 0.20 μm at ×100 objective magnifications. After complete etching about 200 tracks were measured for detector resolution.

2.5. Detector resolution

The track detectors are usually not affected by a high flux of less ionising radiation and thereby offer excellent mass, charge and energy resolution for heavy energetic ions. The energy resolution of a detector is defined as FWHM divided by the mean ion energy (E_m) obtained from a peak centroid. The energy resolution

$$\text{Resolution} = (\text{FWHM}/E_m) \times 100. \tag{1}$$

The FWHM energy resolution and standard deviation in each case are given in table 2.

Table 2. Energy resolution of 350 MeV ^{90}Zr in two different detectors.

Parameters	CR-39	ZnP-glass
Energy (MeV)	350	350
L_{FWHM} (μm)	5.00	5.00
σ_L (μm)	2.124	2.124
E_{FWHM} (MeV)	38.7	54.0
σ_E (MeV)	16.2	22.5
L_m (μm)	51.0	31.0
Resolution (%)	11.06	15.43

σ = standard deviation, FWHM = full width at half maximum, L_m = most probable track length.

2.6. Error analysis

The accuracy in track length measurement is generally found to be ± 0.9 μm leading to an error of ± 1.3 μm in the true track-length measurement. An error of ± 1.3 μm in the true track length leads to a cumulative error of $\pm 4.0\ \mu\mathrm{m\ h}^{-1}$ in the track-etch rate. This corresponds to a percentage error of 2–10 in V_T for the ZnP-glass detector. Accuracy in the measurement of track diameter is ± 0.5 μm which leads to an error in V_G of $\pm 0.03\ \mu\mathrm{m\ h}^{-1}$ for CR-39 and $\pm 0.28\ \mu\mathrm{m\ h}^{-1}$ for ZnP-glass.

3. Results and discussion

3.1. Etch rates

Bulk-etch rates (V_G) for ZnP-glass and CR-39 have been determined using the track-diameter technique [14]. The results are given in table 3. It has been observed that the etching process is faster in ZnP-glass than in CR-39 under a given etching condition (6N NaOH/55 °C). The

Table 3. Values of complete etching time t_c, the bulk-etch rate V_G and maximum etchable track length of 350 MeV ^{90}Zr ions in the two detectors.

	CR-39	ZnP-glass
Etching conditions	6N NaOH/55 °C	6N NaOH/55 °C
t_c (min)	120 ± 4	7 ± 0.25
V_G (μm h^{-1})	0.96 ± 0.03	10.1 ± 0.28
Track length (μm)		
Experimental†	51.0 ± 2.1	31.0 ± 2.1
Theoretical‡	48.94	33.50

† Values representing the most probable track lengths.
‡ From computer code RANGE [13].

time (t_c) corresponding to complete etching of tracks in each case was also determined (see table 3).

The track-etch rate (V_T) at any point on the tracks of ^{90}Zr ions in CR-39 and ZnP-glass were obtained from the slopes of the curves plotted of true track length against etching time. If, for an interval of time Δt, the track length ΔL increases from L_1 to L_2 then the V_T corresponding to the mean track length $L(L_1 + L_2/2)$ is $\Delta L/\Delta t$. For the segment of track ΔL, V_T remained constant.

3.2. Comparison of experimental and theoretical track length

The maximum etchable track lengths of 350 MeV ^{90}Zr in these track detectors have been obtained by applying suitable etching corrections [15] to the observed track lengths. Figure 1 shows the distribution of track lengths of ^{90}Zr in CR-39 and ZnP-glass detectors. The most probable track lengths are found to be 51.0 μm and 31.0 μm respectively. Table 3 lists these experimental values, which were obtained from the computer code 'RANGE' [13], based on the stopping-power equations of Mukherji and co-workers [15–18].

A reasonably good agreement has been observed between the measured and the computed values. This has further supported the earlier views [19–21] that the ranges and track lengths of any heavy ion in any complex medium calculated from the stopping-power equations [15–18] are in good agreement with the experimental values.

3.3. Detector calibration

Here we have calibrated the two detectors (CR-39 and ZnP-glass) with 350 MeV ^{90}Zr ions by two methods, based on the dependence of residual range (R) and total energy-loss rate (dE/dx) on the track-etch rate (V_T) which represents the damage intensity at different points on the tracks. Table 4(a) shows the measured values of V_T at different points on the track with the corresponding residual range (R). Table 4(b) lists the values of V_T as a function of the theoretical total energy-loss rate (dE/dx) for ^{90}Zr ions in the two detectors.

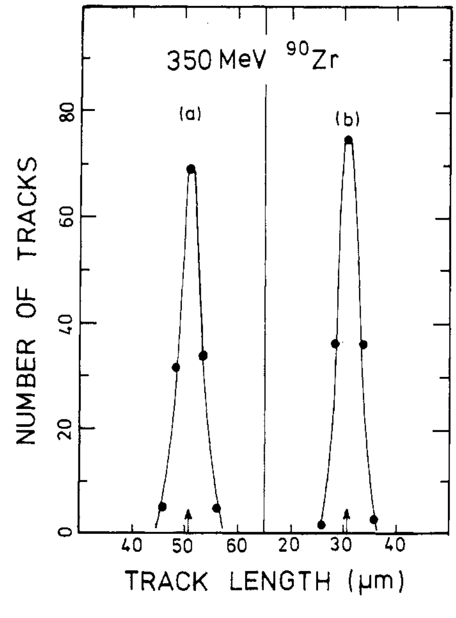

Figure 1. Track length distribution of 350 MeV ^{90}Zr in (a) CR-39 (b) ZnP-glass. Most probable track lengths are indicated by the arrows.

3.3.1. Dependence of V_T on R. It has already been mentioned that the tracks are developed by successive etching. After every etching the residual range is obtained by subtracting the etched track length from the total range. Plots of track-etch rate (V_T) against residual range (R) for ^{90}Zr ions in these detectors are given in figures 2 and 3 respectively.

A linear dependence has been observed between V_T and R for both the track detectors used. The general linear equation shown below represents such a dependence:

$$V_T = a(R) + b \qquad (2)$$

Table 4(a). Values of measured V_T and residual range for 350 MeV ^{90}Zr in CR-39 and ZnP-glass detectors.

Detector	Residual range (μm)	V_T (μm h^{-1})
CR-39	30.00	34.00
	25.00	30.83
	20.00	26.67
	15.00	20.00
	10.00	18.21
	5.00	14.55
ZnP-glass	10.00	200.00
	8.00	150.10
	6.00	120.00
	4.00	100.00
	2.00	66.67
	1.00	42.86

Table 4(b). Values of measured V_T and calculated theoretical energy-loss rate (dE/dx) for 350 MeV ^{90}Zr in CR-39 and ZnP-glass.

Detector	V_T (μm h^{-1})	dE/dx† (MeV mg^{-1} cm^2)
CR-39	34.00	65.00
	30.83	58.00
	26.67	49.00
	20.00	45.00
	18.21	33.00
	14.55	24.00
	13.33	18.00
ZnP-glass	150.10	32.30
	120.00	26.50
	100.00	26.50
	87.50	22.00
	66.67	19.50
	42.86	17.00

†dE/dx calculated from computer code RANGE [13].

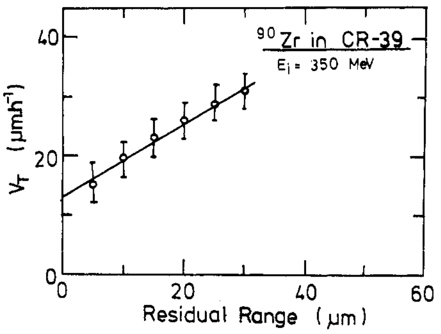

Figure 2. A calibration curve for the CR-39 detector with ^{90}Zr in terms of measured V_T as a function of residual range.

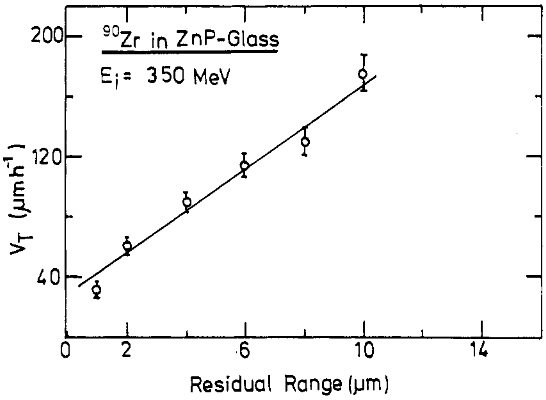

Figure 3. A calibration curve for the ZnP-glass detector with ^{90}Zr in terms of measured V_T as a function of residual range.

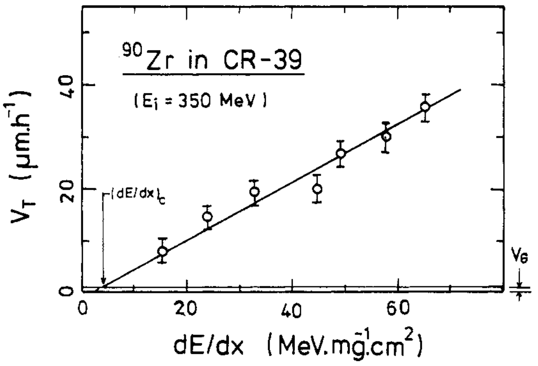

Figure 4. A calibration curve for the CR-39 detector with ^{90}Zr in terms of measured V_T as a function of total energy-loss rate (dE/dx).

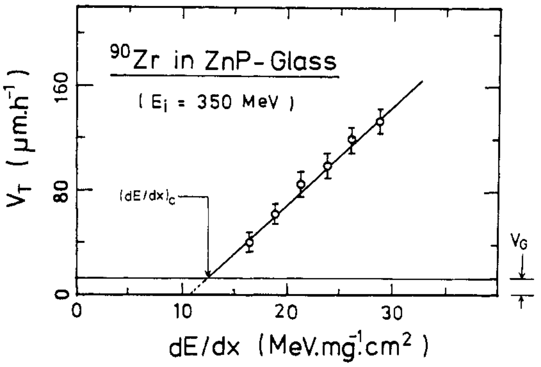

Figure 5. A calibration curve for the ZnP-glass detector with ^{90}Zr in terms of measured V_T as a function of total energy-loss rate (dE/dx).

where V_T is in units of $\mu m\,h^{-1}$ and R is in μm. Coefficients a and b have been evaluated from the slope of V_T–R plots (figures 2 and 3) and are listed in table 5. It was found that V_T–R calibration for ^{90}Zr is valid between 25 and 350 MeV for both.

3.3.2. Dependence of V_T on total energy-loss rate.

Figures 4 and 5 show plots of V_T as a function of total energy-loss rate (dE/dx) for CR-39 and ZnP-glass respectively. A linear dependence is observed between V_T and dE/dx for the two detectors and is expressed by the equation

$$V_T = c(dE/dx) + d \qquad (3)$$

where V_T has the same units as expressed in the previous section and dE/dx is in MeV mg^{-1} cm^2.

The coefficients c and d for both the detectors have been determined from the slope of the corresponding plots of V_T against dE/dx (figures 4 and 5) and are listed in table 6.

The above results are in accordance with the earlier findings [19–21] where a linear dependence of V_T and (dE/dx) has been observed in the case of a few heavy ions in complex media.

In the case of CR-39 and ZnP-glass the critical energy-loss rates $(dE/dx)_c$ have been obtained by linear extrapolation of the V_T–dE/dx curve to the V_G line and are found to be 4.45 and 12.5 MeV mg^{-1} cm^2 respectively.

Table 5. Values of coefficients and validity region of the calibration curve between V_T and residual range (R) for 350 MeV ^{90}Zr ions in two detectors.

	Coefficients		
SSNTD	*a*	*b*	Validity region
CR-39	0.67 ± 0.06	11.70 ± 3.72	>25 MeV
ZnP-glass	16.60 ± 1.60	28.32 ± 4.47	>10 MeV

Table 6. Values of coefficients and validity region of the calibration curve between V_T and energy-loss rate (dE/dx) for 350 MeV ^{90}Zr ions in two detectors.

	Coefficients		
SSNTD	*c*	*d*	Validity region
CR-39	0.44 ± 0.05	−1.00 ± 0.5	entire
ZnP-glass	7.31 ± 0.39	−81.26 ± 9.48	entire

4. Conclusion

The registration and development of latent tracks created by 350 MeV ^{90}Zr in CR-39 and ZnP-glass detectors have been studied in terms of bulk and track-etch rates, complete etching time and maximum etchable track length. Our results show that in 6N NaOH at 55 °C, etching times of 120 ± 4 min and 7 ± 0.25 min are required to completely etch 350 MeV ^{90}Zr tracks in CR-39 and ZnP-glass detectors respectively. Using appropriate etching corrections, the true maximum etchable track lengths of ^{90}Zr in CR-39 and ZnP-glass detectors were found to be $51.0 \pm 2.124\ \mu$m and $31.0 \pm 2.124\ \mu$m respectively. A reasonably good agreement between these values and the corresponding theoretical data from the computer code RANGE has further supported the reliability of the stopping power equations used in the program. One of the significant contributions of this work comes from the fact that a linear dependence of the track-etch rate (V_T) on total energy-loss rate (dE/dx) supports the 'ion-explosion spike' model of track formation. The above correlation and the one between V_T and residual range (R) have been used to calibrate these detectors and to determine the values of track registration thresholds $(dE/dx)_c$ for 350 MeV ^{90}Zr in CR-39 and ZnP-glass detectors.

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