

STUDY OF $^{132}_{54}\text{Xe}$ -ION TRACKS IN MAKROFOL-E POLYCARBONATE PLASTIC TRACK DETECTOR

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SUMMARY : Makrofol-E polycarbonate plastic detector has been exposed to $^{132}_{54}\text{Xe}$ -ion from cyclotron beam at JINR, Dubna, USSR. The bulk etch rate and track etch rate are measured for different etching temperatures and hence the activation energies are determined. The maximum etched track length is compared with the theoretically computed range. The experimental results show that the track etch rate, V_t along the particle trajectory depends on the etching temperature, T but the normalized track etch rate, $V = V_t/V_b$ is independent of T . From the response curve it is evident that V_t depends on energy-loss, dE/dx as well as on T while V depends only on dE/dx and not on T . Moreover, the experimental data are presented for the influence of different annealing conditions on bulk etch rate, track diameter, track etch rate, and etchable range of $^{132}_{54}\text{Xe}$ -ion in Makrofol-E detector.

Key Words : Makrofol-E, accelerated heavy ion, annealing.

INTRODUCTION

In Solid State Nuclear Track Detectors (SSNTDs) paths of individual heavily ionizing charged particles are revealed by selective chemical etching of the radiation damaged material along the particle's trajectory. This track etching technique has been successfully employed in many insulating materials for detection and identification of charged particles (8). We have been interested in measuring particle track parameters in Makrofol-E plastic detector. The track length is one of these parameters providing the range of the particle and hence its energy. The curve of the track etching

rate as a function of the energy deposited along the trajectory of the particle is often called a 'response curve'.

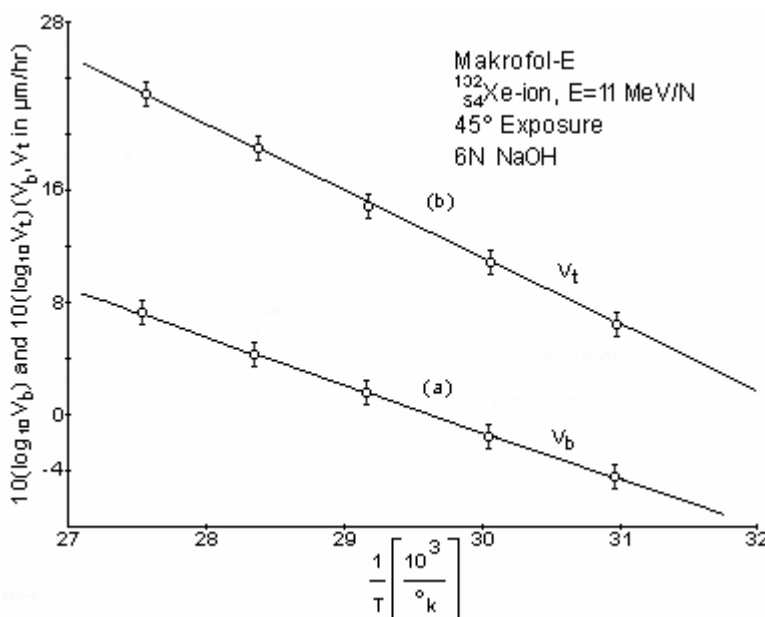
In the present study the bulk and track etch rates are measured for different temperatures. The maximum etched track length is also determined. The dependence of V_t and $V = V_t/V_b$ on etching temperature, T as well as on energy-loss, dE/dx is studied. The experimental results showing the effect of heat treatment on latent tracks before etching are also presented.

EXPERIMENTAL DETAILS

Samples of Makrofol-E exposed to $^{132}_{54}\text{Xe}$ -ion of energy 1.1 MeV/N at angles of 90° and 45° to the

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Figure 1: Variation of bulk etch rate, V_b and track etch rate, V_t with etching temperature for Makrofol-E exposed to 1.1 MeV/N ¹³²₅₄Xe-ion.



detector surface are obtained from JINR, Dubna, USSR. The exposed samples are etched in stirred NaOH (6.00±0.05) N solution. Five different etch bath temperatures namely 50°, 60°, 70°, 80° and 90°C have been employed. The stability of the etching temperature is ±0.5°C. The measurements are taken with an 'Olympus' microscope with a 40x objective and 15x eyepiece. The least count of the eyepiece micrometer is LC=0.215 μm at a magnification of 900x.

RESULTS AND DISCUSSION

Effect of temperature on bulk etch rate, V_b

The bulk etch rate, V_b is determined by the following two methods (2,4,6,7):

- (i) Gravimetric method
- (ii) Track diameter method

The bulk etch rate is determined for 50°, 60°, 70°, 80° and 90°C. Both the methods give the same results within the limits of uncertainty. This shows the isotropic

etching character of Makrofol-E plastic detector. The results are presented in Figure 1(a). The straight line indicates the exponential dependence of V_b on etching temperature and can be expressed by the relation,

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

where A is a constant, E_b is the activation energy for bulk etching, T is the etchant temperature in absolute scale and k is the Boltzmann constant. From the slope of the straight line, the value of E_b is calculated to be

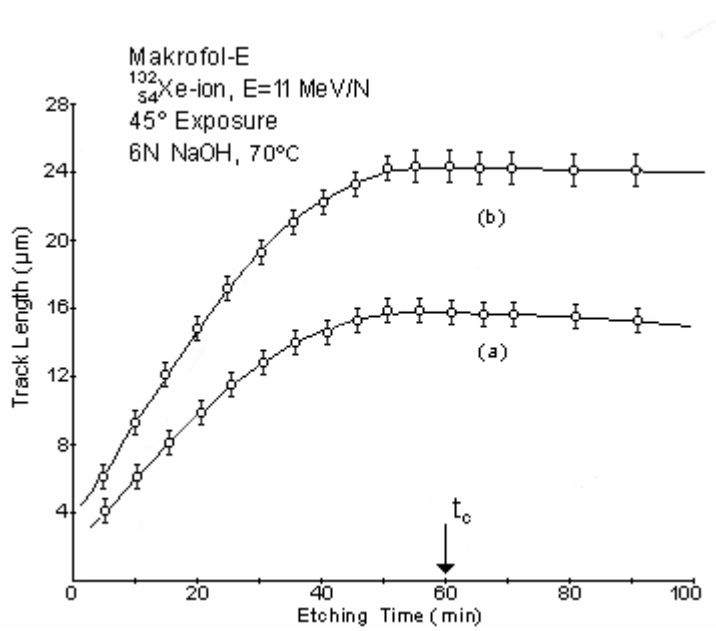
$$E_b = (0.72 \pm 0.06) \text{ eV.}$$

This value is in good agreement with that reported in literature (2,3).

Variation of track diameter with etching time

The samples exposed to ¹³²₅₄Xe-ion at an angle of 90° are etched in 6N NaOH solution at several tem-

Figure 2: Variation of (a) observed track length and (b) corrected track length of ¹³²₅₄Xe-ion in Makrofol-E with etching time.



peratures. The track diameters are plotted as a function of etching time. The curves (not shown) reveal a linear relationship between track diameter and etching time for different temperatures.

Effect of temperatures on track etch rate, V_t

When the samples of Makrofol-E detector exposed to ¹³²₅₄Xe-ions at an angle of 45°C are etched in 6N NaOH solution we observed the appearance of conical tracks. The projected track lengths are measured and the corrected projection length, l_p is determined. The detailed procedure is discussed elsewhere (1,2,4-7). The true track length, L (the length from the original surface to the terminal end of the track) is determined by the relation (1,2,4-7):

$$L = \frac{l_p}{\cos\delta} + \frac{V_b t}{\sin\delta} - V_b(t - t_c) \quad (2)$$

where δ = angle of incidence, $V_b/\sin\delta$ = the surface

etching correlation, $V_b(t-t_c)$ =the over-etching correction, t_c =the time required to etch the tracks up to the point where they stop (etched until the track ends become round).

The track etch rate, V_t is calculated by the relation

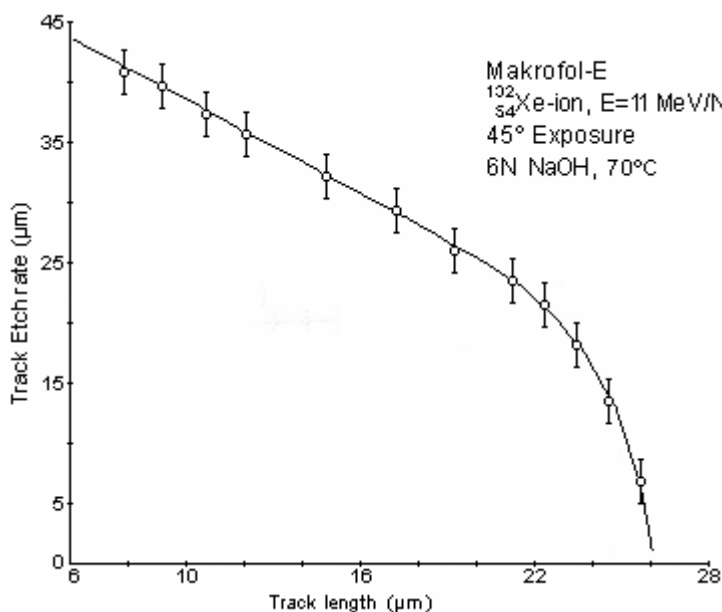
$$V_t = \frac{\Delta L}{\Delta t} \quad \dots\dots (3)$$

where ΔL is the track length increase in etching time Δt .

Samples of Makrofol-E detector exposed to ¹³²₅₄Xe-ion at an angle of 45°C are etched in 6N NaOH solution at 70°C. The plot of the variation of and l_p with etching time is shown in Figure 2. Using Ex. (3), the values of V_t at different points on the track are obtained from Figure 2(b). Figure 3 shows the variation of V_t with track length, L for 70°C. Clearly V_t decreases with penetration depth.

Following the same procedure the plots of V_t versus L are also drawn for 50°, 60°, 80° and 90°C. These plots are very similar in nature to that seen in Figure 3 but are

Figure 3: Variation of track etch rate with track length of ¹³²₅₄Xe-ion in Makrofol-E.



not shown. From these plots, the track etch rate, V_t corresponding to a particular track length, L ($=18 \mu\text{m}$ in this case) is determined for different temperatures. The effect of etching temperature on V_t is shown in Figure 1(b). The rise in track etch rate with etching temperature, T (in absolute scale) is again found to be exponential and may be expressed by the relation,

$$V_t = B \exp(-E_t/kT) \dots \dots \dots (4)$$

where B is a constant and E_t is the activation energy for track etching. The value of E_t is calculated to be

$$E_t = (0.70 \pm 0.04) \text{ eV.}$$

The values of $V = V_t/V_b$ for different temperatures have been calculated from the experimentally determined V_t and V_b values. We found that for Makrofol-E, there is no change of V (i.e. track registration sensitivity) with etching temperature between 50° and 90°C .

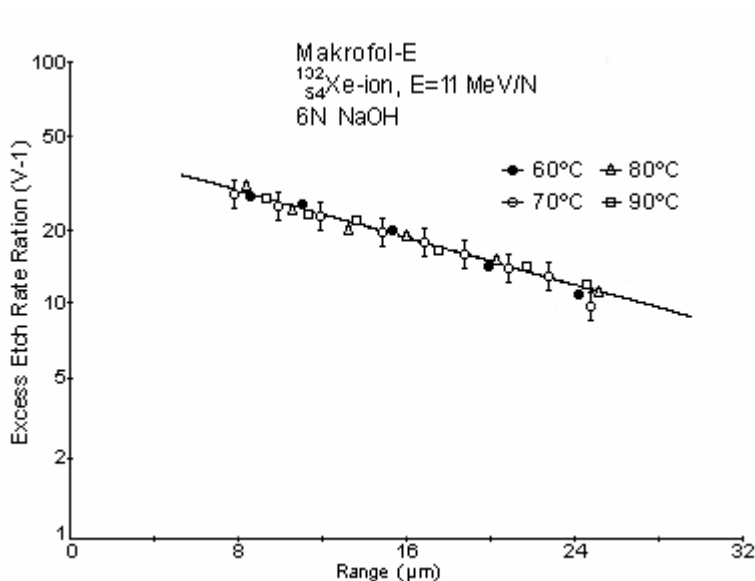
By noting $\sin O = V_b/V_t$, we can conclude that the

cone angle of ¹³²₅₄Xe-ion track in Makrofol etched in 6N NaOH solution remains constant between etching temperatures 50° and 90°C .

Range of ¹³²₅₄Xe-ion in Makrofol-E

The average length of maximum etched tracks (etched until the tips of the tracks become round) is calculated to be $L = (25.50 \pm 1.04) \mu\text{m}$. To compare this maximum etched track length with the theoretical range of ¹³²₅₄Xe-ion in Makrofol-E, the stopping power and range equations of Mukherji and Nayak (11) have been used. A computer program is made using these equations and with the help of computer the range of ¹³²₅₄Xe-ion in Makrofol-E (C_{16}, H_{14}, O_3 and $\rho = 1.20 \text{ gm/cm}^3$) is computed. The computer lists the energy-loss $\delta E/dx$ and the penetration dept (i.e. range) starting from the initial ion energy down to zero at intervals of $dE = (0.01 \text{ MeV})$. The theoretically computed range is found to be $R = 26.25 \mu\text{m}$. The maximum etched track length agrees with the calculated range to better than 3%.

Figure 4: Plot of excess etch rate ratio as a function of range of ¹³²₅₄Xe-ion in Makrofol-E for different temperatures.



The evaluation of the track etch rate along the particle trajectory

A simple relation between the etch rate ratio, V and the particle range, R has been found for ¹³²₅₄Xe-ion. In Figure 4 data of the excess etch rate ratio, (V-1) are plotted versus the range, R of ¹³²₅₄Xe-ion in a semi-logarithmic scale for different temperatures. It is concluded that the track etch rate, V_t along the particle trajectory depends on the etching temperature but the normalized track etch rate, V=V_t/V_b is independent of etch bath temperatures. The solid line is the best fitted line to the experimental points. A reasonable representation of the line can be given by the relation of the form (13,14) :

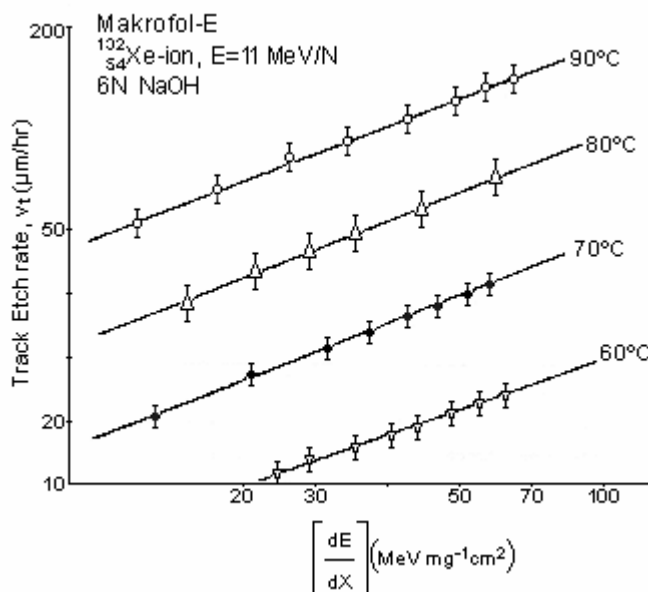
$$V=1+\exp (-AR+B)$$

where A and B are fitting parameters. With the help of computer these values are calculated. They are A=0.058 and B=3.842.

The 'response curve'

From the computer output, the plot of energy-less, dE/dx versus penetration depth has been drawn (not shown). The variation of the track etch rate, V_t with track length is shown in Figure 3. Combining these two figures, the 'response curve' [(dE/dx) vs V_t] is plotted on a double-logarithmic paper (Figure 5) for etch bath temperature of 70°C. The (dE/dx) vs V_t curves for different etching temperatures are also presented in the same figure. It is immediately apparent from Figure 5 that the track etch rate, V_t depends on dE/dx as well as on etching temperature. In Figure 6 the normalized track etch rate, V=V_t/V_b are plotted against (dE/dx) in a linear diagram for four different etching temperatures. It is evident that all our data normalized in this way belong to the same curve within the accuracy of the measurements. It is concluded that the normalized track etch rate depends only on (dE/dx) and not on the etching temperature.

Figure 5: Variation of track etch rate with energy loss for $^{132}_{54}\text{Xe}$ -ion tracks in Makrofol-E for different temperatures.



Effect of annealing on bulk etch rate, V_b

Unexposed samples of Makrofol are annealed at different temperatures for 30 minutes. The samples are then etched simultaneously in 6N NaOH at 70°C. The removed layers of the sheets are measured with micro thickness gauge having least count = 0.5 µm. It is observed that there is no change in the bulk etch rate even at 190°C. Even at this annealing temperature the sheets are satisfactorily elastic. Lasting deformations are observed from 175°C upwards. Annealing times longer than 30 min lead to gradual deterioration of the surface qualities of the plastic, which is undesirable in their application as track detectors.

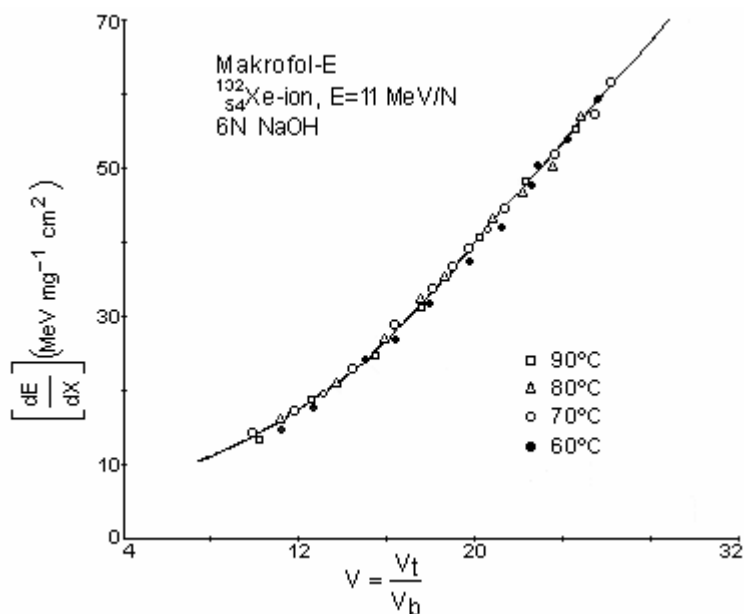
Effect of annealing time and temperature on track diameter

Samples of Makrofol exposed vertically to $^{132}_{54}\text{Xe}$ -ions are annealed at various temperatures for 10 minutes. From our earlier experiences and the prescription of Khan and Durrani (9) for polycarbonate

plastics, the annealing time is chosen as 10 minutes. The annealed samples are etched in 6N NaOH at 70°C. The etchpit diameters are measured as a function of the removed layer. The results are shown in Figure 7. The inset in Figure 7 gives a plot of the track diameters after removing a 10 µm thick surface layer as a function of annealing temperature. Thus to eradicate $^{132}_{54}\text{Xe}$ -ion tracks completely, 10 min of annealing must be applied at 195°C in Makrofol-E detector.

Exposed samples of Makrofol are annealed at 150° and 165°C for different lengths of time. Figure 8 shows the effects of annealing time on the etchpit diameters of $^{132}_{54}\text{Xe}$ -ions entering vertically, after removing a 10 µm thick surface layer of the individual detector samples. It can be observed that each curve consists of a steeply and a slowly falling component. It is apparent from the figure that a part of the radiation-damaged region produced by $^{132}_{54}\text{Xe}$ -ion is already eradicated at the given temperature by short annealing, however in the more stable region persistent after the treatment, no

Figure 6: Plot of normalized track etch rate, V_t/V_b versus dE/dx for ¹³²₅₄Xe-ion tracks.



considerable change is brought about even by a significant extension of annealing time. At higher temperature of annealing (165°C in Figure 8), the slowly falling component of the curve starts early. Since the measurements at other temperatures for ¹³²₅₄Xe-ions display a similar tendency, they are not shown.

Effect of annealing temperature on track etch rate, V_t

Experiments are conducted to show the decrease in V_t of ¹³²₅₄Xe-ions tracks. The ratio of track and bulk etch rates ($V=V_t/V_b$) is evaluated for different temperatures from the initial slopes, S of the curves shown in Figure 7 using the relation (5,10,12,13) :

$$V = \frac{1 + 0.25S^2}{1 - 0.25S^2}$$

Then this etch rate ratio is plotted against the annealing temperature as shown in Figure 9. It is observed that V_t decreases with annealing temperature.

Effect of annealing temperature on diameter distribution

Samples of Makrofol exposed vertically to ¹³²₅₄Xe-ions are annealed for 10 min at 150°C. The areas of annealed and unannealed samples are determined precisely to obtain the track density. The annealed and unannealed samples are etched simultaneously in 6N NaOH at 70°C. Figure 10 shows the diameter distribution of ¹³²₅₄Xe-ion tracks in Makrofol after removing 12 μm thick surface layer of the individual detector. The experiments reveal that after annealing, not only the track density is reduced, but also the diameters of the etched ¹³²₅₄Xe-ion tracks are diminished as a result of lower etching velocity in the damage trail.

Effect of annealing temperature on maximum etchable range

Makrofol-E detector samples exposed to 1.1 MeV/N ¹³²₅₄Xe-ions at an angle of 45°C with respect to the detector surface are annealed at different tempera-

Figure 7: Relationship between track diameter and removed layer for $^{132}_{54}\text{Xe}$ -ion tracks in Makrofol-E annealed for 10 min at various temperatures.

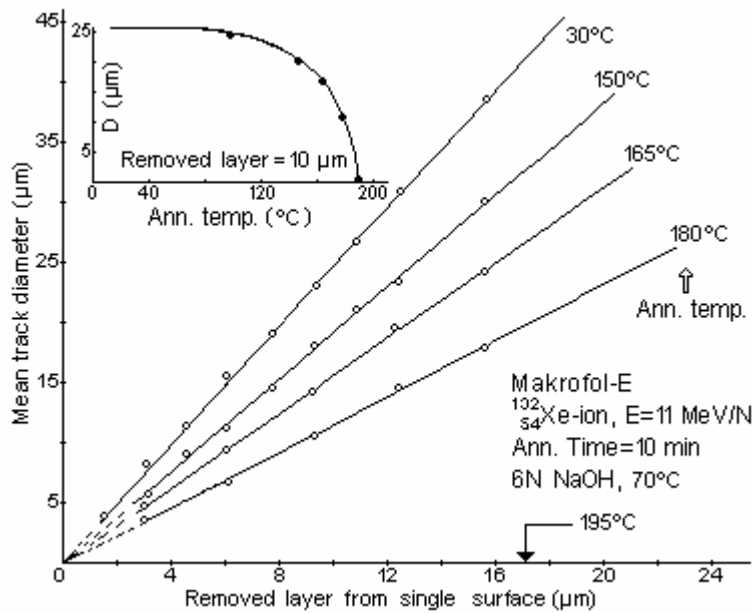


Figure 8: Relationship between track diameter and annealing time for $^{132}_{54}\text{Xe}$ -ion tracks in Makrofol-E detector annealed at 150°C and 165°C.

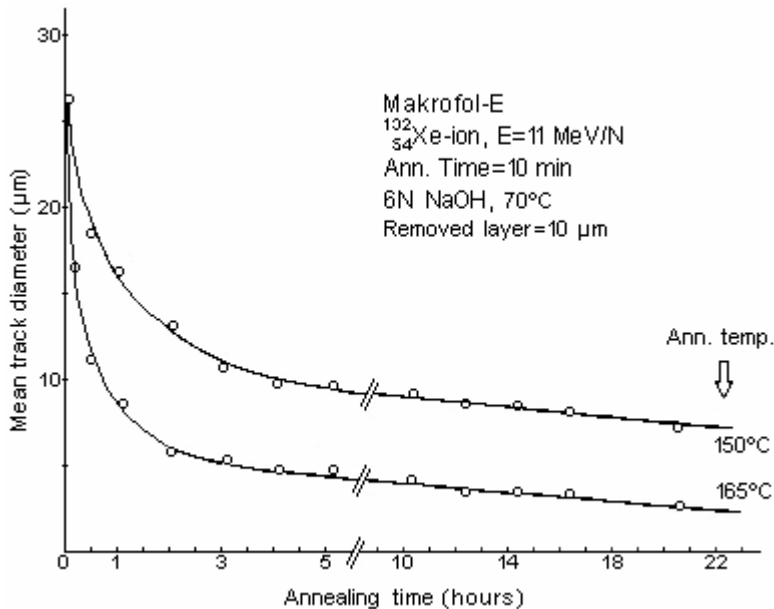


Figure 9: Etch rate ratio, $V=V_t/V_b$ as a function of annealing temperature for $^{132}_{54}\text{Xe}$ -ion in Makrofol-E detector.

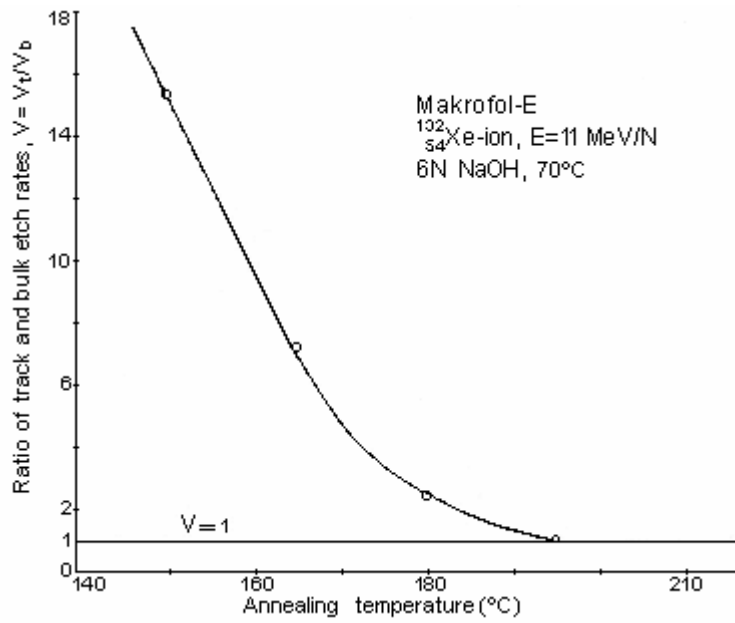


Figure 10: Diameter distribution of unannealed and annealed $^{132}_{54}\text{Xe}$ -ion tracks in Makrofol-E plastic detector.

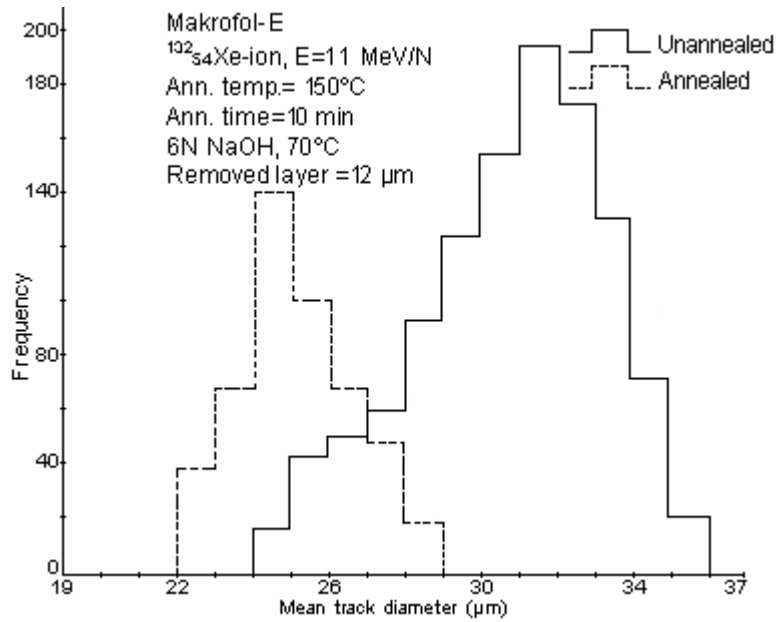
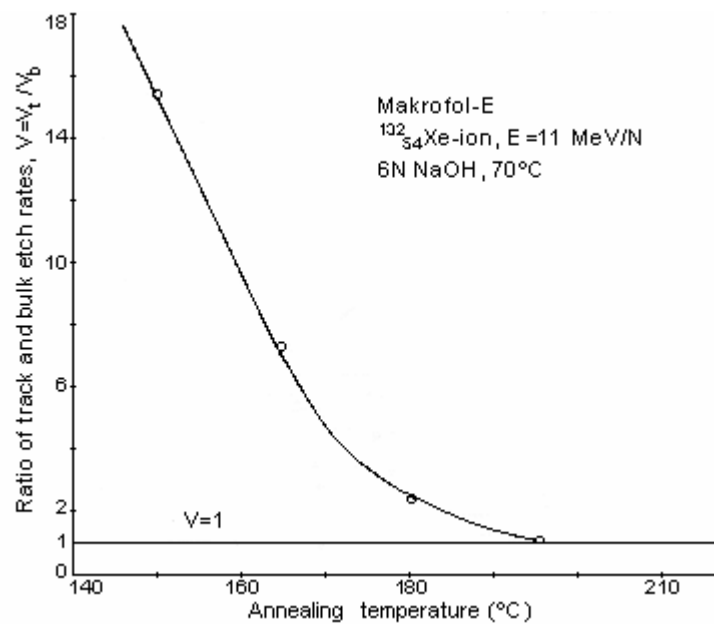


Figure 11: Variation of maximum etchable track length (i.e. range) of $^{132}_{54}\text{Xe}$ -ion tracks in Makrofol-E with annealing temperature.

tures for 10 minutes. The samples are then etched simultaneously in 6N NaOH at 70°C until the track ends become round. The maximum etched track lengths are determined. The effect of annealing temperature on maximum etchable length (i.e. range) of $^{132}_{54}\text{Xe}$ -ions in Makrofol-E is shown in Figure 11. It is clear from the figure that the track length decreases by the application of heat. Again it is noted that the tracks are completely erased out when annealed for 10 min at 186°C. From the results shown in Figures 7 and 11 it can be concluded that the vertical tracks are more stable than oblique tracks and require higher temperature for their complete erasure.

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