# ANNEALING EFFECTS ON TRACKS IN MAKROFOL DETECTORS (SSNTDs)

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#### Abstract

A change of fission track density and track length in Makrofol with different annealing temperature and annealing time was investigated. Ten pieces of (1 cm x 1 cm) Makrofol detectors each was contacted with <sup>252</sup> Cf source for 1 hour. Among them, two pieces were unannealed. Four pieces of Makrofol with fission tracks were annealed at 60° C and the other four pieces were annealed at 100°C for 30 min, 60 min, 90 min and 120 min respectively. After that the annealed and unannealed detectors were etched in 6.25N NaOH at 60°C for 75 min. The fission fragment tracks were counted and the tracks length were measured with the aid of an optical microscope. The average tracks densities of detector were calculated at different temperature and various time. It was found that the tracks length and tack density decrease with the increasing of annealing time and annealing temperature.

Keywords : Makrofol, SSNTDs, unannealed, annealed

#### Introduction

Solid state nuclear track detectors (SSNTDs) have been used in different areas of science such as nuclear physics, particle physics, cosmic ray physics, environmental physics, and archaeology. Polycarbonate Track Detector is a commonly used Solid State Nuclear Track Detector (SSNTD) to identify the fission fragment. Makrofol, a polycarbonate material, is a particular group of thermoplastic polymers. It is available in three types which are Makrofol N, KG and Makrofol SKG blue. Makrofol N and KG are not available in suitable thickness below 20  $\mu$  m. Makrofol SKG blue is available in suitable thickness of 2.0  $\mu$ m and 6.0  $\mu$ m. Makrofol polycarbonates are prepared from diphenylol-alkanes, of which the commonest is 2,2- diphenylol - propane or Bisphenol A (BPA) with structural formula as shown in figure 1(a)and (b).

Makrofol-N (white in colour) is a bisphenol-A polycarbonate with a chemical composition of  $C_{16}H_{14}O_3$  known as a good industrial material with high impact resistance and dimensional stability over a broad temperature range.



Figure 1 (a) Photograph of Makrofol-N detector (b) The structural diagram of Makrofol

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When ionizing particles like alpha particles and fission fragments pass through a nonconducting material, like crystal, polycarbonate, etc., damaged sites known as latent tracks occur along their paths. Polymers composed of large molecules and the damages form as a result of breaking of the molecular bonds.

The annealing is supposed to start with recombination of ions and electrons to form atoms at interstices and later diffusion of atoms towards the damaged zone. The diffusion takes place under stress. One of the advantages of this process is to eliminate the pre-existing background track signal. And annealing temperature must be carefully applied since overheating causes some structural changes in the detector material and in extreme conditions may even disfigure it.

If an SSNTD is chemically etched after exposure to radon and fission products, tracks caused by the radiation will enlarge. These tracks can then be counted with an ordinary optical microscope.

The present paper deals with the study of thermal annealing with track density and track length of <sup>252</sup>Cf fission fragments using unannealed and annealed Makrofol-N detectors.

#### **Experimental Procedure**

#### Materials and methods

In the present work, Makrofol-N polycarbonate plastic was used as fission track detector. Dependence of fission track length and track density of Makrofol on annealing time and temperature was studied. The block diagram of experimental procedure of Makrofol is shown in figure (2).



Figure 2 The block diagram of experimental procedure of Makrofol

## Irradiation on Makrofol with <sup>252</sup>Cf

Californium <sup>252</sup>Cf fission source was used for irradiation of the makrofol detectors in the present work. Its diameter is 25 mm and its thickness is 0.5 mm. The active area is a diameter of 10 mm and the source activity is 1.02  $\mu$ Ci. The nuclide <sup>252</sup>Cf disintegrates with a probability of 3.09 % by spontaneous fission. The fission fragments have fission energies 80 MeV and 104 MeV. Its half-life is 2.64 years. The energy of emitted a particles are 6.1184 MeV, 6.076 MeV and 5.977 MeV. The fission rate is about10<sup>3</sup> fission per  $\mu$ Ci with fission fragments energies ranging from 80 MeV to 104 MeV.

A sheet of Makrofol ( $C_{16}$  H<sub>14</sub> O<sub>3</sub>) detectors were cut into small pieces with dimension of 1 cm x 1 cm in each piece. Each piece of detector was irradiated for 1 hour with <sup>252</sup>Cf sources in contact under the atmosphere. The schematic diagram of detector irradiation was shown in figure (3).



Figure 3 Detector irradiation with <sup>252</sup>Cf source

#### Annealing technique with different temperature

After irradiation, the tracks of fission fragments were formed in the pieces of Makrofol samples. Among them, four pieces each was heated in a muffle furnace at  $60^{\circ}$  C for 30 min, 60 min, 90 min and 120 min respectively. Then they were cooled under the atmosphere at the room temperature. Other four pieces of irradiated Makrofol detectors each individual was annealed at 100<sup>o</sup>C from 30 min to 120 min respectively. After annealing all the detectors were cooled down at the room temperature.

#### **Preparation of the Etchant**

6.25 N NaOH solution was prepared for etching all the annealed and unannealed detectors. To obtain 6.25 N NaOH solution, 25 g of NaOH pellets (99% purity) were put into 100 ml measuring cylinder. Then distilled water was poured on the NaOH pallets in the measuring cylinder and stirred with a glass rod, until all NaOH pellets were dissolved. The distilled water was added to get 100 ml solution. After that, the solution was poured into a 100 ml glass beaker. Figure (4)



Figure 4 Preparation of 6.25 N NaOH

#### Etching of the detectors

The solution of NaOH (6.25 N) in the beaker was heated on a stove with temperature controller. The process of etching and microscopic observation are repeated till the optimum condition of tracks and the maximum track length became invariant with furture etching. When the temperature reached at 60 °C, it has no invariant condition, the annealed and unannealed detectors of Makrofol were put into the beaker for 70 min. All samples were held at the same depth in the etchant solution. During etching, the temperature was kept 60 °C with an accuracy of  $\pm 1$  °C. After etching, the etched detectors were collected with plastic sieve with handle. Then, the detectors were washed under the running water until the surfaces of detectors were cleaned from etchant. Finally the detectors were dried with filter paper. Figure (5).



Figure 5 The schematic diagram of chemical etching

#### Track Visualization with optical Microscope

After doing the etching process, the tracks visualization, track photographing, track counting and measuring the length of tracks were done by using optical microscope (Nikon Eclipse 50 i ) at a magnification of 40 x and 100 x to get the fission track length and number of fission tracks.(Figure 6)

The microscope was attached with DS camera and camera control unit to take microphotograph of detectors. The number of tracks on the detector was recorded view by view by changing the vertical and horizontal position of detector.



Figure 6 optical microscope (Nikon Eclipse 50 i ) with DS -5M camera

The average track density on the detector was calculated from the following equation.

Average Track density = 
$$\frac{\text{no of truck}/_{\text{view}}}{\frac{\text{area}}{/_{\text{view}}}}$$

After that, the standard deviation of track densities and track length were calculated from the following equation.

#### **Standard Deviation's equation**

$$SD = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (x_i - \bar{x})^2}$$
Where, N = no of views  
x = no of tracks per area  
 $\bar{x}$  = average tracks per area  
SD = standard deviation

#### **Results and Discussions**

After observing the variation of track lengths and track densities under optical microscope attached with digital camera, the following results for each annealing condition have been expressed.

The average track density due to different annealing temperature at 60°C and 100°C are shown in Table (1) and Table (2). The curves using data from Table (1) and (2) are shown in Figure (7) and (8).

The average track length due to different annealing temperature at 60°C and 100°C are shown in Table (3) and Table (4). The curves using data from Table (3) and (4) are shown in Figure (9) and (10).

Figure (11) and Figure (12) showed the comparison of average fission track densities and track length in makrofol detectors after annealing at 60°C and 100°C for different annealing time.

The photographs of Makrofol detector with fission track of annealing time for 30 min and 60 min at annealing temperature 60°C and 100°C were illustrated in Figure (13) to Figure (16).

For Makrofol detectors with fission track, it is found that the average track density and average track length decrease with increasing annealing temperature and annealing time. It may be due to the repairing of broken molecular chain in the Makrofol as annealing proceeds.

Table 1Average fission track densities in Makrofol SSNTDs for after annealing at 60°Cfor different annealing time

Sr. No	Annealing	Annealing time	Average track densities
	temperature (°C)	(min)	(track/mm <sup>2</sup> )
1.	-	0	$115.92 \pm 3.52$
2.	60	30	$110.20 \pm 3.67$
3.		60	$107.75 \pm 3.89$
4.		90	$99.18\pm2.03$
5.		120	$91.43 \pm 3.90$

# Table 2 Average fission track densities in Makrofol SSNTDs for after annealing at100°C for different annealing time

Sr. No	Annealing temperature (°C)	Annealing time (min)	Average track densities (track/mm <sup>2</sup> )
1.	-	0	$82.44 \pm 2.06$
2.	100	30	$78.36 \pm 2.32$
3.		60	$71.83 \pm 1.75$
4.		90	$65.30 \pm 1.23$
5.		120	$60.41 \pm 1.37$

Table 3 Average fission track length in Makrofol SSNTDs after annealing at 60°C

Sr. No	Annealing time (min)	Average track length (µm)
1.	0	$6.24 \pm 1.47$
2.	30	$5.36 \pm 1.67$
3.	60	$4.92 \pm 1.44$
4.	90	$3.80 \pm 1.23$
5.	120	$2.76 \pm 1.65$

Sr. No	Annealing time (min)	Average track length (µm)
1.	0	$3.84 \pm 0.83$
2.	30	$3.20 \pm 1.52$
3.	60	$2.82\pm0.89$
4.	90	$2.22 \pm 1.00$
5.	120	$1.89 \pm 0.81$

Table 4 Average fission track length in Makrofol SSNTDs after annealing at 100°C



**Figure 7** The variation of average track densities in Makrofol SSNTDs after annealing at 60°C for different annealing time.



Figure 8 The variation of average track densities in Makrofol SSNTDs after annealing at 100°C for different annealing time.



Figure 9 The comparison of average track lengths due to different annealing time for annealing temperature at 60°C and 100°C in makrofol (SSNTDs)



Figure 10 The variation of average track lengths with different annealing time at 100°C in Makrofol (SSNTDs)



Figure 11 The comparison of average fission track densities in Makrofol SSNTDs after annealing at 60°C and 100°C for different annealing time.



Figure 12 The comparison of average track lengths due to different annealing time for annealing temperature at 60°C and 100°C in Makrofol (SSNTDs)



**Figure 13** Photomicrograph of fission tracks in Makrofol detector after annealing at 60°C for 30 min



Figure 14 Photomicrograph of fission tracks in makrofol detector after annealing at 60°C for 60 min



Figure 15 Photomicrograph of fission tracks in makrofol detector after annealing at 100°C for 30 min



**Figure 16** Photomicrograph of fission tracks in Makrofol Detector after annealing at 100°C for 60 min

### Conclusion

From the results, the average densities of unannealed Makrofol are 115.92  $\pm$  3.52 track/  $mm^2$  and 82.44  $\pm$  2.06 track/mm^2 . And the average track lengths are 6.24  $\pm$  1.47  $\mu m$  and 3.84  $\pm$  0.83  $\mu m$ . It was found that the average density and average track length of unannealed samples were more than that of annealed samples at different temperature and time by comparing data. There is an annealing effect on tracks in both Makrofol. The annealing of the damage trails at elevated temperature presumably occurs via the movement of molecular fragments with a Makrofol.

It was concluded that the tracks length and tack density decrease with the increasing of annealing time and annealing temperature.

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