

AFM investigation of the track structure in polymer films

D.L.Zagorski , A.I.Vilensky, G.S.Zhdanov*, N.V. Pervov, B.V.Mchedlishvili

**Shubnikov Institute of Crystallography, Moscow, Russia, Leninski pr., 59, 117333,
tel.:(007-095) 135-99-71, fax: (007-095) 135-10-11, E-mail: mic@eimb.ru**

*** Institute of Physics and Power Ingeneering, Obninsk, Russia**

INTRODUCTION

Some types of polymers are known as excellent track detectors. These polymers can be used both for accelerated ions and fission fragments detection. The structure of tracks and their thermal stability are of great interest.

Irradiation with high-energetic particles produces tracks in polymers –the cylindrical areas of polymer destruction. It is known that heat treatment of irradiated polymer strongly affect on these tracks and the result of such treatment depends on its temperature [1].

The effect of acceleration of further etching (sensitization) took place for the treatment temperatures lower than glass-transition temperature (70-80 $^{\circ}$ C for PET). For example in [2] it was found that annealing of the irradiated films in the temperature interval 30-80 $^{\circ}$ C hardly changed the etching kinetic because of desorption of air components from the tracks.

The annealing of irradiated polymer at the temperatures higher than glass-transition temperature leads to disappearing (“healing”) of latent tracks (LT) or to reduction of their detectability. This effect supposed to caused by displacements of macro-molecules fragments and decontamination of active centers. [1,3].

At the same time tracks were found to have compound structure: different radial areas were discovered [4,5].

EXPERIMENTAL

Biaxially oriented poly(ethylene terephthalate) (PET) film with thickness 10 μ m was irradiated with Xe ions (energy 1 MeV/nucleon and fluence 10^9 ions per sq.cm.) and Bi ions (3 MeV/nucleon and fluence 10^9 ions per sq.cm) at U-400 cyclotron (Joint Institute of Nuclear Research, Dubna).

Thermal treatment (annealing) of irradiated polymers was carried out in air atmosphere in the temperature interval from 50 $^{\circ}$ C to 180 $^{\circ}$ C during 1 h.

Irradiation by U^{235} fission fragments was carried out at nuclear reactor BR-10 (Institute of Physics and Power Ingeneering, Obninsk). The packet of 15 ultra thin (2.5 μ m) closed-packed polymer sheets was used for fission-fragments irradiation.

The atomic-force microscope (AFM) Solver P-47 (NT-MDT, Russia) operated in resonance regime (“tapping”, in air, at room temperature) was used for investigation of samples surface. The unit was completed with silicon cantilevers (NT-MDT, frequencies 150-350 kHz and a tip curvature radius 10 nm).

RESULTS AND DISCUSSION

Ions tracks

AFM image of the irradiated polymer demonstrates the small holes with diameter 7 nm, which are the track entries at the film surface: their surface density corresponds to the ion irradiation fluence. The thermal treatment of the polymer samples (at the temperatures 50 $^{\circ}$ C – 180 $^{\circ}$ C during 1 h) highly changed the surface character: the large cavities with diameter 30 nm appeared instead of small ones. It may be connected with polymer structure changing in volume.

Bi- irradiation, produced more intensive radiation damage of polymer. That gave us possibility to investigate peculiarities of track structure in details. Fig.1 demonstrates the damaged area: hillock with diameter more than 100 nm with the crater (diameter 10-15 nm) on its top. During further annealing the following process took place: the hillock diameter does not changed up to the temperatures 160 $^{\circ}$ C, while their height first increased (20-25 nm at the temperature 130 $^{\circ}$ C- see Fig.2) and then decreased during further annealing. At the temperature 180 $^{\circ}$ C only small hillocks are visible.

The most interesting fact is the thermal behavior of the crater on the top of hillock- these craters are detected up to the temperatures 70 $^{\circ}$ C and then disappeared. This fact is in good correlation with the peculiarities of etching of irradiated polymers: the etching speed is maximal up to the temperature 70-80 $^{\circ}$ C- it may be connected with “healing”, annealing of the track channel.

Fission-fragment tracks

The irradiation chamber had the temperature approx. 100 $^{\circ}$ C, so, the process of annealing took place simultaneously with the irradiation. The irradiation of the “packet” of many thin polymer sheets gave us possibility to investigate “the deep” structure of track. It is known that U²³⁵ nuclei usually disintegrate into two unequal parts with different energies (for example- Sr and Xe with energies approx. 115 MeV and 85 MeV-). According to this two types of radiation defects were found at the surface of outside sheet. Fig.3 demonstrate the surface radiation damage for lower-energy fragment (possible- Xe). It must be mentioned, that in this case the damaged area is much larger than in the case of Xe-ions with the same energy- it is connected with different electrical charge of Xe (approx. 8 for accelerated ions and approx. 25 in the case of fission fragments).

The sheets from the 1t to 5 th were tested and monotonic decrease of the defect size was found. This work is in progress now.

CONCLUSIONS

It can be supposed that the track in irradiated polymer has complicated structure and consists of different zones. Polymer annealing at the temperatures higher than glass-transition temperature results not to simply “healing” (which means slowly disappearing of track), but to complex process of destructed areas changing. This process leads to increasing of density of track areas, additional cross-linking which increase chemical stability of the track areas (to compare with non-annealed). These areas, in their turn, consists from different zones, probably connected with the zonal structure of tracks in irradiated polymer.

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Fig. 1

Fig. 2

Fig. 3