

# Investigation of heavy ion tracks in polymers by transmission electron microscopy

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Bulk samples and thin sections of polyethylene terephthalate (PET) and polyimide (PI) were irradiated with Se and Pb ions of 11.4 MeV/u at the UNILAC. The creation of latent tracks and related structural changes were studied by means of transmission electron microscopy (TEM) using a 200 kV Philips CM200-UTW microscope (point resolution 0.17 nm). Due to relatively similar electron densities within organic polymers, staining helps to increase the imaging contrast and improve the radiation resistivity under the electron beam.

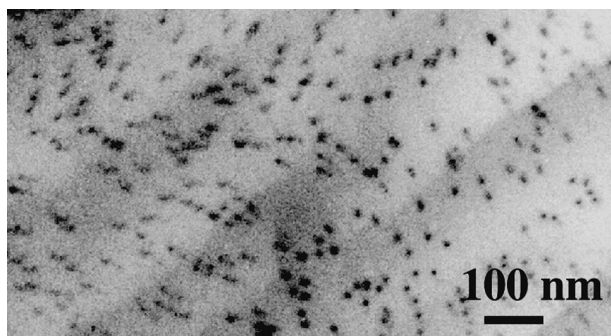


Fig. 1. TEM image of an originally 50 μm thick PET foil irradiated with Se-ions ( $6 \times 10^{10} \text{ cm}^{-2}$ , 900 MeV). The dark spots are the ion tracks decorated after irradiation by  $\text{OsO}_4$ .

Typical stains such as  $\text{OsO}_4$  or  $\text{RuO}_4$  contain elements with high atomic number and diffuse preferably into the amorphous regions of the polymer thus enhancing the contrast of different structural regions. We investigated several sample preparation techniques, e.g. staining in vapor phase or aqueous solution, applied at different stages before and/or after ultra-microtomy and ion irradiation.

Fig. 1 shows the TEM micrograph of a PET sample irradiated as an initially 50-μm thick foil. Subsequently, the film was stained (aq.  $\text{OsO}_4$ , 7 days) and cryo-sectioned perpendicular to the ion trajectories. The resulting ultrathin sections were stained again (2 h,  $\text{OsO}_4$  vapor). The tracks are visible as dark spots indicating that the stain is preferentially accumulated in the amorphous track regions. The mean diameter is  $12 \pm 3$  nm. The areal density is in good agreement with the ion fluence.

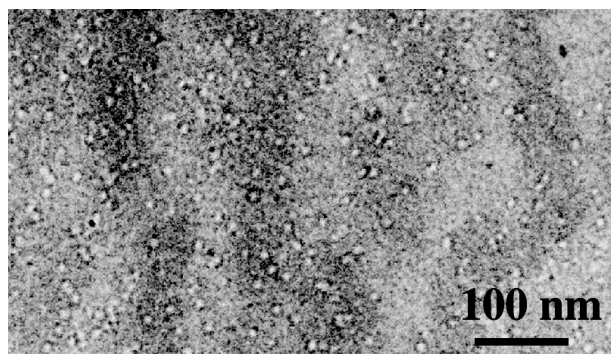


Fig. 2. TEM image of ultrathin section of PET, pre-stained (30 min, aqueous  $\text{RuO}_4$ ) prior to irradiation with Pb-ions ( $5 \times 10^{10} \text{ cm}^{-2}$ , 2370 MeV).

The situation is quite different if pre-stained polymer samples are irradiated as thin sections (fig. 2). Tracks appear as light

features of reduced contrast with a mean diameter of  $9 \pm 1$  nm. The stain and probably even the polymer material are expelled from the track region by sputtering and outgassing. Under a tilted angle, the number of tracks is twice as large as expected because we image two spots per track, namely the impact region on the front and back side of the sample. The track cylinders in the bulk are visible, but the contrast fades rapidly during observation.

Compared to PET, the electron-beam resistance of PI is better, allowing imaging at even higher magnifications. Fig. 3 presents a single track in a pre-stained (aq.  $\text{RuO}_4$ , 2 h) and then irradiated 50-μm thick PI film. After ultramicrotomy, the thin section was stained again ( $\text{OsO}_4$  vapor, 2 h). Note that in this case, tracks are imaged as bright regions although staining was performed after ion irradiation (cf. PET, Fig. 1). Since both, matrix and track regions, are amorphous, the selectivity of the post-staining process is apparently not high enough for preferential track decoration. In the matrix around the track, but not inside, the phase contrast of small crystalline areas of Ru or Os compounds can be recognized.

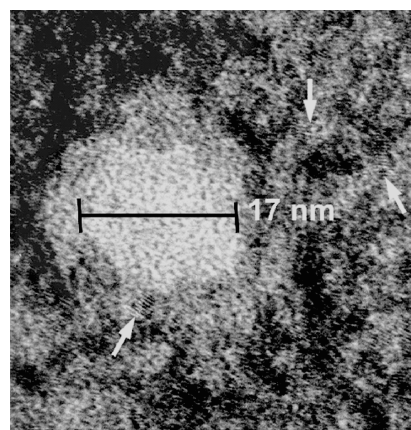


Fig. 3. High resolution TEM image of a single Pb-ion track in PI. The arrows indicate small crystalline areas in the track-surrounding matrix.

The track size observed here is in reasonable agreement however, slightly larger than diameters deduced earlier using other techniques such as small-angle x-ray scattering or IR-spectroscopy [1]. Also similar TEM studies on polyethylene [2] and on PI without staining [3] reported about 30% smaller track diameters. Finally, it should be mentioned that the precise determination of the track size in polymers using TEM is difficult due to the damage induced by the electron beam. The tracks may shrink or increase during imaging, making a direct comparison of different observations rather problematic.

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- [2] J. Vetter, G. H. Michler, I. Naumann, Radiat. Eff. Def. Sol. 143 (1998) 273.
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