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In situ small-angle x-ray scattering measurements of ion track etching in polymers

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When a highly energetic heavy ion passes through a target material, the damaged region left in its wake often exhibits preferential chemical etching over the undamaged material. This etch-anisotropy can be used to create very high aspect ratio channels (pores) of up to tens of microns in length, with pore diameters as small as several nanometres. Membranes formed by this method are ideal for many advanced applications including ultra-filtration, bio- and medical sensing, nano-fluidics, and nano-electronic devices. The shape of the etched pores can be cylindrical, conical or double conical, depending on the etching conditions. One major advantage of the technique is the ability to generate arrays of pores that are highly parallel with extremely narrow size distributions.

The aims of this research are to develop a detailed understanding of the track etching process and the etching kinetics in polymers by performing *in situ* small angle x-ray scattering (SAXS) measurements during the etching process. The SAXS measurements were carried out at the Australian Synchrotron in Melbourne, Australia. Investigating the influence of etching parameters and pore areal density on nano-pore formation enables the controlled fabrication of nano-pore membranes with size and shape-specific pores. For our experiments we used 12 µm thick foils of PET and 20 and 30 µm thick polycarbonate (PC) foils, irradiated with 2 GeV ^{197}Au-ions at the GSI UNILAC in Darmstadt, Germany. The irradiated material was subsequently etched in diluted sodium-hydroxide (NaOH) at several concentrations and temperatures. The etching was conducted in a custom-built sample environment while performing the SAXS measurements in transmission mode to determine the track etch rate as a function of etch time. These *in situ* scattering images were analysed using a batch fit method to determine the pore size as a function of etching time. An example of a transmission SAXS scattering image of cylindrical pores in PC is shown in Fig. 1. The results of the study indicate that the track etching behaviour is strongly influenced by temperature and concentration of the etchant, whereas the pore areal density only has a small effect on the etch rate. This allows the calculation of activation energies for radial etching of PET and PC depending on their pore areal densities. The etch rates for PC are largely linear, however PET seems to have two etch rates indicating a damaged halo.

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