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

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Introduction

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. 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 of nano-pores in polymers by performing *in situ* small angle x-ray scattering (SAXS) measurements during the etching process.

Methods

SAXS measurements were carried out at the Australian Synchrotron in Melbourne, Australia. Investigating the influence of etching parameters on nano-pore formation enables the controlled fabrication of nano-pore membranes with size and shape-specific pores. For our experiments, we used 12 and 19 µm thick foils of PET and 20 and 30 µm thick polycarbonate (PC) foils, irradiated with 2.2 GeV 197Au ions and 1.1 GeV 131Xe ions at the GSI UNILAC in Darmstadt, Germany. The irradiated material was subsequently etched in sodium-hydroxide (NaOH) at several concentrations and temperatures. The etching was conducted in a custom-built *in situ* sample environment while performing the SAXS measurements in transmission mode. These measured scattering images were analysed using a batch fit method to determine the shape, pore size, size distribution and surface roughness as a function of the etching conditions.

Results

All etch rates for PC and PET are linear above pore radii of 20 nm. Using an Arrhenius law, the activation energies for radial etching of PET and PC were determined. The activation energy in PET (1.42 eV) is almost doubled compared to PC (0.78 eV). Nano-pore dimensions in PET are more polydisperse compared to PC, due to an increased polycrystallinity of the material. During etching, the size distribution decreases with increasing etching temperature and increasing etchant concentration. PET shows a larger surface roughness compared to PC. This roughness is attributed to a gel layer at the interface between polymer and etchant.

Conclusion

*In situ* SAXS measurements of ion track etching in polymers are feasible and can yield unprecedented details about the etching process. Very detailed information about the evolution of the pore characteristics are obtained. The results of the study allow to precisely determine the effect of temperature and concentration of the etchant on the pore formation process. New insights into the etching process such as the influence on the pore size distribution and properties of the gel layer have been found.

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