ESRF	**Experiment title:** In-situ observation of plastic zone formation during crack propagation in polymer adhesion	**Experiment number:** SC646
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Report:

The craze morphology created with the double cantilever beam test method (DCB) was investigated with small angle scattering using a micro focus. The DCB is a technique to estimate the interface toughness between two polymer plates. For a measurement a razor plate is pushed between them (see figure 1) and from the resulting crack length the calculation of the interface toughness is possible. The energy is not only put in the construction of the crack between the plates but also in the formation of a plastic zone. This means that at the tip of the crack crazes perpendicular to the interface inside each plate appear. Details of the deformation are not quite well known up to now.

Our model system consists of polymethylmethacylate (PMMA) of different molecular weights (88k, 134k and 191k). The samples were prepared by annealing two PMMA plates on top each other, applying a slight pressure. Different annealing times have been used to install different interface toughnesses via interdiffusion of polymer chains at the interface. The typical dimension of the crack tip as well as of the crazes in the plastic zone is on the order of a few micrometer only. Thus investigations yielding a position sensitive signal are restricted to extremely small beam diameters. Using a common large beam size averages out the complete information by a convolution of the signal with the background from the unperturbed sample volume. For that reason we used a beam with a diameter of 12μm and a wavelength of 0.948Å. With a two-dimensional detector (CCD camera) structures up to 50nm were resolved.

For the first time a spatial resolved mapping of the crack tip and the accompanied plastic zone in a polymer model system under DCB geometry was recorded. A typical mapping covered an area of 200µm * 150µm (y * z see figure 1) in steps of 10µm in each direction including the crack tip and the plastic zone.

Depending on the beam position different small angle scattering pattern have been observed. In figure 2 three different examples are shown. The micrograph in figure 2 shows the corresponding spatial positions of the beam (marked with white circles). The crack and its tip is visible as the black horizontal and vertical line. In figure 2a the scattering signal is dominated by the bulk background, which is not subtracted to visualize its shape. Compared to figure 2b, which shows a scattering resulting from the plastic zone and the bulk background, the intensity is very small. Therefore we have a marked contribution of the sample features introduced by the DCB method, in contrast to previous experiments utilizing larger beam sizes.

Depending on the interface toughness we observed different scattering pattern. For example also intensity distributions similar to those observed in ductile aluminium, which was investigated previously. These pattern are dominated by single or multiple finger like streaks in a wide angular distribution. Other intensity distributions show clear evidence for the presence of small equidistant structures, e.g. fibrils. With respect to these complicated scattering features a detailed data analysis is in progress.

The expected results will have important implications on further experiments using more complex polymeric materials including crystallization or phase separation.

Fig. 1
Schematic drawing of the SM-SAXS experiment relative to sample and crack. The circles show the beam positions.

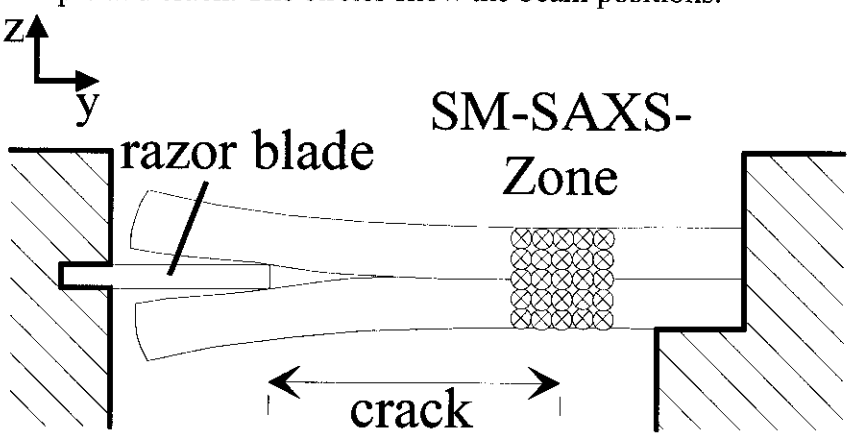

Fig. 2
The micrograph below shows the end of a crack. The interface is so tough that the crack already snaped up- and downwards out of it. The circles show the positions of the beam and the images on the right the corresponding SAXS images. The sample consists of PMMA 191k plates that had been annealed on top of each other for 1700 min at a reference temperature of 130°C.