

**Experiment title:**

Toughening mechanism in high impact polystyrene studied by means of ultra-small angle X-ray scattering

Experiment number:

SC1514

Beamline:

ID2

Date of experiment:

from: 30.11.04 to: 02.12.04

Date of report:

22.02.2005

Shifts:

6

Local contact(s):

Dr. Peter Bösecke

*Received at ESRF:***Names and affiliations of applicants (* indicates experimentalists):**

Dr. Yongfeng MEN*, Dr. Jens RIEGER, Jörg MALCHUS*

BASF Aktiengesellschaft

Polymer Physics

GKP/S G201

67056 Ludwigshafen, Germany

Report:

Pure polystyrene (PS) is brittle at room temperature which limits its range of applications. To overcome this problem small soft rubber particles are introduced into the PS matrix to form high impact polystyrene (HIPS) [1]. Despite of its wide range of applications, the underlying toughening mechanism in HIPS is still under discussion [2-6]. When stretched, HIPS always shows a dilatational behavior accompanied by strain whitening due to crazing in the matrix and/or cavitation in the rubber phase. Crazes are cracks with fibrils spanning between the crack walls. The experiments carried out and described in this report were aimed to elucidate the sequence of crazing and cavitation during continuous stretching of HIPS by performing online synchrotron small angle X-ray scattering at ID02 at ESRF.

Experiments:

A commercial HIPS was used in the experiments. It contains “salami”-like rubber particles of 7 μm diameter. The total rubber content is 9.1%.

Samples were cut from a compression molded plate of 1mm thickness into a form indicated in figure 1.

The dimension of the sample is also given in figure 1. Online stretching experiments were conducted using a portable stretching device mounted at the beamline. The samples were stretched at a constant speed of 20 $\mu\text{m/s}$. The force required for elongation was recorded simultaneously with the scattering data so that each scattering pattern

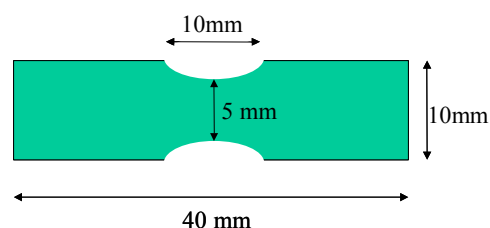


Figure 1. The dimensions of HIPS sample for online stretching

corresponds to a certain point on the force elongation curve. Scattering data were collected in one second intervals with an exposure time of 100 ms.

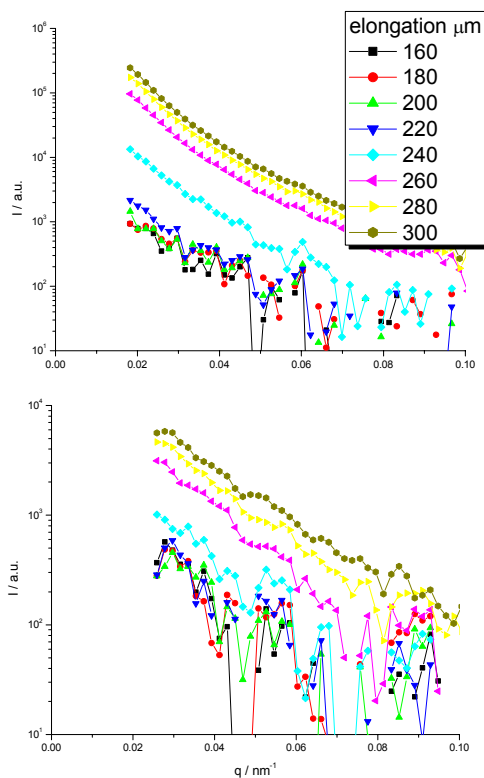


Figure 2. one dimensional intensity distribution along stretching direction (top) representing the scattering cavities and perpendicular to the stretching direction (bottom) representing the scattering from the craze fibrils.

The sample to detector distance was 10 m. To overcome the problem of possible reflection of X-rays at the craze bulk interface, the stretching device was tilted by 10° . The force elongation curve (not shown) exhibits at first a strong increase in loading force when stretching. After an elongation of about $240 \mu\text{m}$, yielding occurs accompanied by a drastic strain softening. Further stretching only leads to a slight increase in loading force. It was observed that after the initial stage of the stretching curve, no further changes in the scattering data occurred. One dimensional scattering intensity distributions measured in the early stages of the craze/cavity formation along and perpendicular to the stretching direction are given in figure 2. Since at small elongations below $200 \mu\text{m}$, no excess scattering is seen, only data of elongations ranging from 160 to $300 \mu\text{m}$ are shown in figure 2.

First of all, one finds a much stronger scattering intensity along the stretching direction as soon as yielding takes place (elongation larger than $240 \mu\text{m}$). The possible reason for this is the occurrence of debonding between the rubber particles and the PS matrix producing cavities of plate-like structure which scatter X-rays intensively along the meridian direction.

The data in figure 2 also provide information about the sequence of crazing and cavitation. When the sample was stretched to $220 \mu\text{m}$, the blue curve in the top plot exhibits a slight increase in intensity compared to the scattering intensity of the sample that was deformed to a lesser degree. However, the bottom plot tells us that there is no excess scattering along the equatorial direction at an elongation of $220 \mu\text{m}$ implying that no crazes are produced yet. It is thus evident that cavitation occurs earlier than crazing in the present case. A detailed analysis of this phenomenon is planned for a publication that is in preparation.

1. Bucknall, C. B. in *The Physics of Glassy Polymers*, Haward, R. N.; Young, R. J., editors; Chapman & Hall: London, 1997.
2. Kambour, R. P. *Macromol.Rev.* **1973**, 7, 1.
3. Paredes, E.; Fischer, E. W. *Makromol.Chem.* **1979**, 180, 2707.
4. Bubeck, R. A.; Buckley, D. J.; Kramer, E. J.; Brown, H. R. *J.Mater.Sci.* **1991**, 26, 6249.
5. Salomons, G. J.; Singh, M. A.; Bardouille, T.; Foran, W. A.; Capel, M. S. *Macromolecules* **1999**, 32, 1264.
6. Ijichi, Y.; Kojima, T.; Suzuki, Y.; Nishio, T.; Kakugo, M.; Amemiya, Y. *Macromolecules* **1993**, 26, 829.