Fracture Behaviour of Polymers - In situ Investigations in the ESEM

Fracture Behaviour of Polymers - In situ Investigations in the ESEM. A tensile stage mounted in an ESEM allows the simultaneous recording of the stress–strain curves and the progress of crack propagation. Thus the global macroscopic values can be correlated with the evolution of features at the crack tip, strongly influenced by the microscopic structure of the material.

Introduction
The conventional tensile tests suffer from one major disadvantage: The stress–strain curves are global macroscopic values, averaged over the whole material. Thus generally little or no information can be gained relating to specific details of the microstructure of the material. But additional information can be provided by operating a tensile stage in the specimen chamber of an ESEM (Environmental Scanning Electron Microscope). A great depth of focus and a great range of magnification enable the recording of the crack propagation with sharp images of both the whole crack tip and special features of it simultaneously with the stress–strain diagrams. Thus the evolution of features like tip blunting, fibril formation, the spalling of surface layers etc., all of which are dependent on the microstructure of the material, can be directly observed and correlated with the macroscopic data. Because of its small depth of focus, light microscopy would not suffice for this type of investigations.

Materials, Instrumentation
Mainly V-notched Charpy impact test specimens of iPP (isotactic polypropylene) and EPR (ethylene propylene rubber)- modified iPP with different sizes of the EPR-particles were investigated. The materials differed additionally in their MFR (melt flow rate) and the type (both α- and β-nucleated) of crystallinity. Higher MFR-values correspond to lower ductility. Generally the specimens were additionally pre-cracked with a razor blade immediately before the tensile test. The samples were characterised both before and after the tensile tests by light microscopy, scanning electron and transmission electron microscopy.
The tensile tests were carried out by use of an **M5000 Tensile Stage** from Deben mounted in an ESEM Quanta 600F from FEI (Fig. 1). The jaw speed can be varied in the range of 0.02 to 4 mm/min. On the tensile stage a cooling / heating platform (from Gatan) can be mounted additionally. The microscope itself can be fully automated by homemade scripts, thus enabling a standardisation of the recording of the tests, e.g. by using the same sequences of magnifications for all specimens. The tensile tests can be recorded by videos, thus enabling the unattended operation and documentation in case of long-lasting experiments with low jaw speeds.

**Microstructure – Macroscopic Behaviour**

The evolution of the structures at the tip of the crack is, of course, dependent on both the jaw velocity and the microstructure of the material. This is clearly visible from Fig. 2, where series of images from videos of three different tensile tests are cut out at the same strains, marked by the **vertical lines** in Fig. 3. An increase in the jaw velocity enforces a stronger tip blunting and an earlier tearing apart of the fibrils formed very early at the tensile tests (series A and B). Actually polymers show a **more ductile** behaviour at lower jaw speeds. Additionally, the appearance of shear lips (see arrow in series A) points immediately out that the skin and the bulk material of the specimen might have different ductilities. This is confirmed by Fig. 4A, a LIMI micrograph of the cross section of the specimen. The bright spots indicate the distribution of the crystalline parts of the material, the **spherulites**. The spherulites are spheriform aggregates of chainfolded fibrillar or lamellar primary crystallites [1]. The utmost layer is nearly amorphous, the next layer comprises few rather small spherulites, followed by the bulk with its high density of big spherulites. The presence of at least three components of the specimen with different ductility can easily be extracted from the videos. Other information is available too: e.g. at which strain the spalling of the skin is starting. But it would be rather difficult, if not impossible, to get this information from only the stress–strain diagrams in Fig. 3, which are a superposition of all the contributions of these three components and also include the changes caused by the different jaw speeds. It
could also be demonstrated that kinks in the stress–strain diagrams, like the one marked by an arrow in Fig. 3, are caused by the rupturing of a greater bundle of fibrils. The series of images in Fig. 2 also demonstrates the formation of wave-like structures at higher strains. These are also caused by the inhomogeneous microstructure and are a result of the spalling of the two surface layers, as is demonstrated by the micrograph in Fig. 4B, showing a region of such a structure imaged with much higher magnification. Polypropylene can crystallize in several structures. The two most important are the α- (monoclinic) and the β-spherulites (hexagonal). The series of images from a β-nucleated specimen in Fig. 2B resembles that of Fig. 2C. This similarity in the behaviour is also confirmed by the stress–strain diagrams in Fig. 3. But the videos also demonstrate, that the density of the fibrils is obviously lower in case of the specimen 2C. The only difference between the specimens 2A and 2B is the replacement of at least part of the α-spherulites by β-spherulites. Thus the β-spherulites show a more ductile behaviour than the α-spherulites. This is also corroborated by literature [2], [3].

Polymers and the glass point
Below a specific temperature, the glass transition temperature, which is itself dependent on the type of polymer, the amorphous content of polymers becomes brittle. Thus in case of polymer blends one could ‘turn off’ one of the components and keep track of the behaviour of the ductile remainder of the polymer blend. This could be especially interesting in case of the analysis of relaxation mechanisms. Work of this type of investigations is in progress.

Resume
The ESEM facilitates the investigation of nonconductive specimens, which makes an in situ tensile testing of polymers possible. This provides new insight in the fracture processes of polymers and the correlations between the macroscopic stress–strain curves and the microscopic morphology.

Literature

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