In situ investigations in the ESEM Advances in materials science

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The environmental scanning electron microscope ESEM enables the investigation of specimens without the need of an electrically conducting surface. Thus, even *in situ* investigations of non conducting specimens are possible. With *in situ* investigation modules like Peltier cooling stages, tensile stages, heating stages and an *in situ* ultramicrotome 3View^{TM} from Gatan, Inc. a great variety of different *in situ* investigations can be performed. Yet, not only the use of the commercially available modules leads to new results, but especially the combination of different methods, the development of new experimental setups or even the development of automated experiments using homemade scripts leads to results which cannot be obtained easily otherwise.

In situ tensile testing of polymers gives the opportunity to correlate macroscopic parameters like the characteristic of a specimen and the microscopic deformation mechanisms gained by imaging the microstructures developing at the crack tip of the sample. Several new methods have been developed for the investigation of polymers: cryo tensile testing of polymers in the ESEM [1], *in situ* tensile testing of peel films [2] and *in situ* tensile testing of polymers coupled with acoustic emission analysis. For this purpose several special mountings were designed for highly specialized investigations. Fig. 1 shows a stress-displacement curve of a polypropylene specimen and micrographs of the crack tip, which were recorded at the displacements marked by vertical lines.

In situ ultramicrotomy with the system 3View^{TM} in the ESEM combines automated serial sectioning of samples and imaging of the specimen´s block face after each cut (Fig. 2a). This method, known as serial block-face scanning electron microscopy (SBFSEM), was developed especially for applications in life science [3], but is also applicable in materials science. Fig. 2b shows a 3D-visualization of the crack region of a polypropylene specimen after a tensile test up to 25% of yield stress [4] (compare Fig. 1a). For this purpose a block was cut out of the tensile specimen and stained with ruthenium tetroxide. Different phases of the material can be discerned (blue: ethylene propylene rubber (EPR) particles in the polypropylene matrix) and the field of cracks (yellow) can be investigated. This demonstrates that a combination of several *in situ* methods can lead to exciting new information about a material.

The ESEM equipped with a *heating stage* enables the direct observation of changes of the morphological structure of the specimen with high magnification and high depth of focus during heating [5]. The pole piece of the microscope is protected against the heat by a heating shield. Two different designs for this heating shield are possible: one is mounted at the wall of the specimen chamber and inserted between the pole piece and the heating stage and an additional voltage can be applied to it to avoid image distortion caused by thermic electrons. The other one is directly mounted on the heating stage. The latter arrangement leads to a shorter working distance, thus better resolution and the application of a conventional BSE-detector should be possible. BSE detectors are much more sensitive to compositional changes

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than the normally used gaseous secondary electron detector. This higher sensitivity is useful for the observation of the onset of scale formation at metals during heating experiments.

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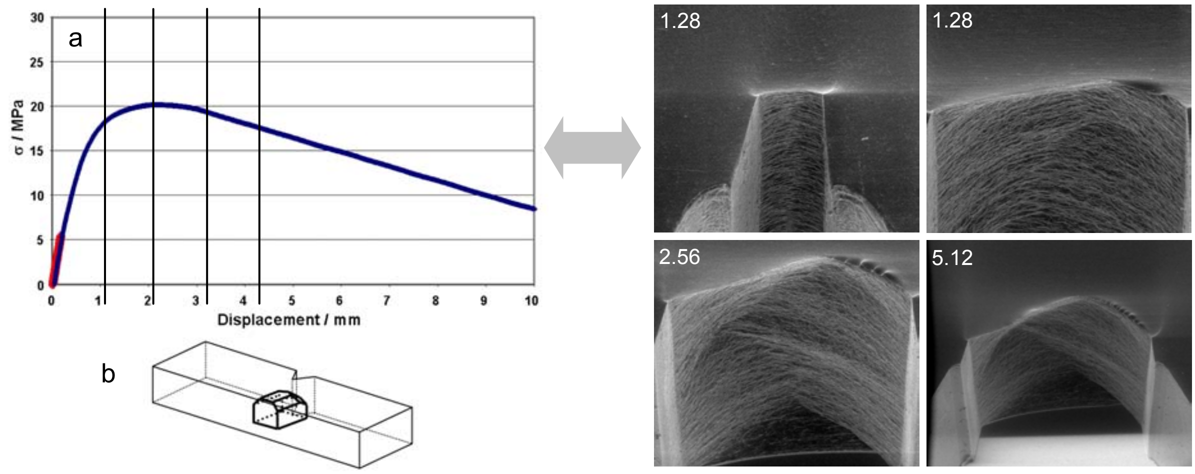

Figure 1. a: Stress-displacement diagram of a v-notched polypropylene specimen. The vertical lines mark displacement values at which the micrographs at the right (SE image, low vacuum mode, ESEM; numbers: image widths in mm) were imaged. **b:** Schematic of the geometry of the specimen and the location of the block cut out for characterization with ultramicrotomy.

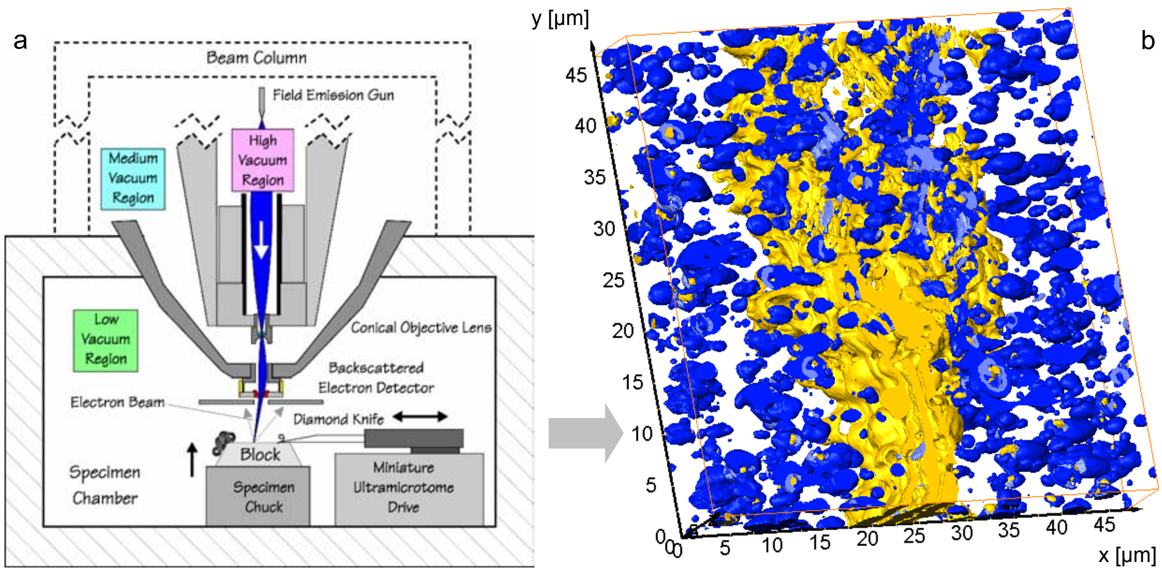

Figure 2. a: Schematic of the *in situ* ultramicrotome 3View^{TM} from Gatan, Inc. **b:** 3D-visualization (program: AMIRA 3.1^{TM}) showing the crack region of the polypropylene specimen of Fig. 1, which was tested with the tensile stage in the ESEM up 25% of the yield stress (see the red curve in Fig. 1). Blue: particles in the polymer matrix; yellow: cracks and voids.

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