Polymer fracture – What can the 3D reconstruction of crack regions tell us about their microscopic fracture mechanisms?

M. Nachtnebel[1], C. Mayrhofer[2], A. Zankel[1,2], P. Pölt[1,2]

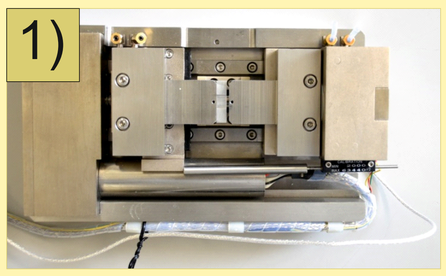
[1] Institute of Electron Microscopy and Nanoanalysis, NAWI Graz, Graz University of Technology, Steyrergasse 17, 8010 Graz, Austria
[2] Graz Centre for Electron Microscopy, Steyrergasse 17, 8010 Graz, Austria

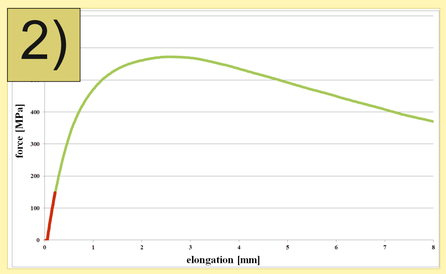
Introduction

The fracture behaviour of polypropylene (PP) modified with either ethylene propylene rubber (EPR) or linear low-density polyethylene (LLDPE) was investigated by serial block-face scanning electron microscopy (SBEM) [1]. Prior to the SBEM process tensile tests were performed (A) [2], subsequently a part of the fracture region was extracted and prepared to fit the requirements of SBEM (B). Subsequently the SBEM image stacks have to be processed (filtered and segmentated) to enable the 3D reconstruction of different features of interest (C). Finally the labeled features can be 3D reconstructed and the impact of tensile tests on different samples compared to each other can give new insights in a more detailed manner (D).

Methodology and results

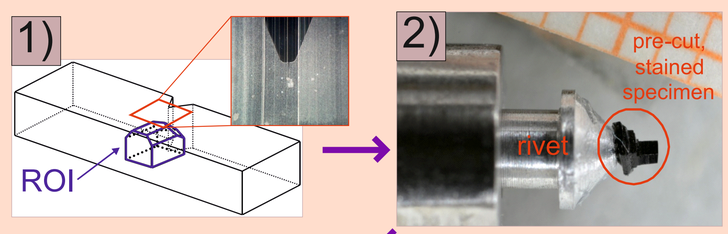
A Tensile test

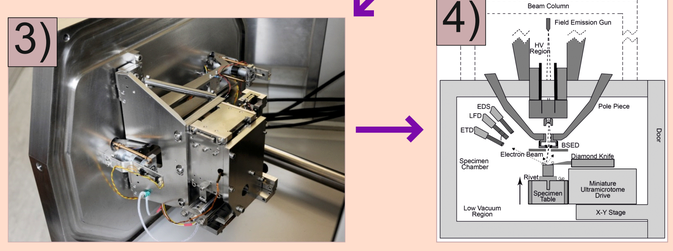




Samples of iPP modified with either EPR or LLDPE particles of different sizes were subjected to tensile tests 1); the tests were stopped at a predefined force, of around 25 % or 50 % yield for the hereby presented results 2).

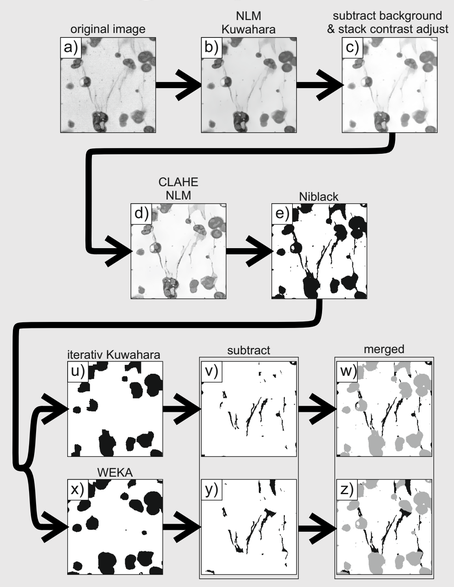
B Sample preparation & SBEM





A part of the fracture region was extracted 1) and prepared by conventional microtomy. After staining with RuO_4 and glueing the sample on an Al-rivet 2) the sample is mounted in the *in situ* ultramicrotome 3) to perform SBEM 4).

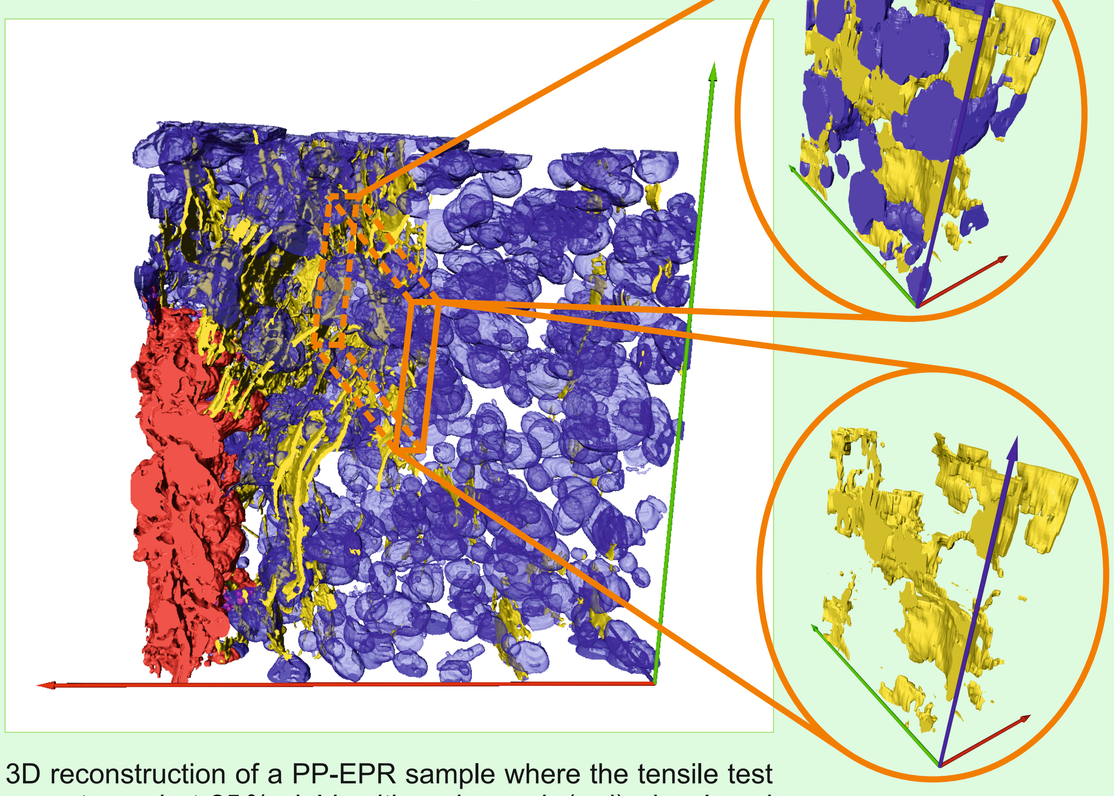
C Image processing



For the image segmentation it is neccesary to initially filter the obtained images (b) – d)) to enhance their contrast. Subsequently, one of the two segmentation algorithms is used to enable the 3D reconstruction of the different phases (u) – w) or x) – z)).

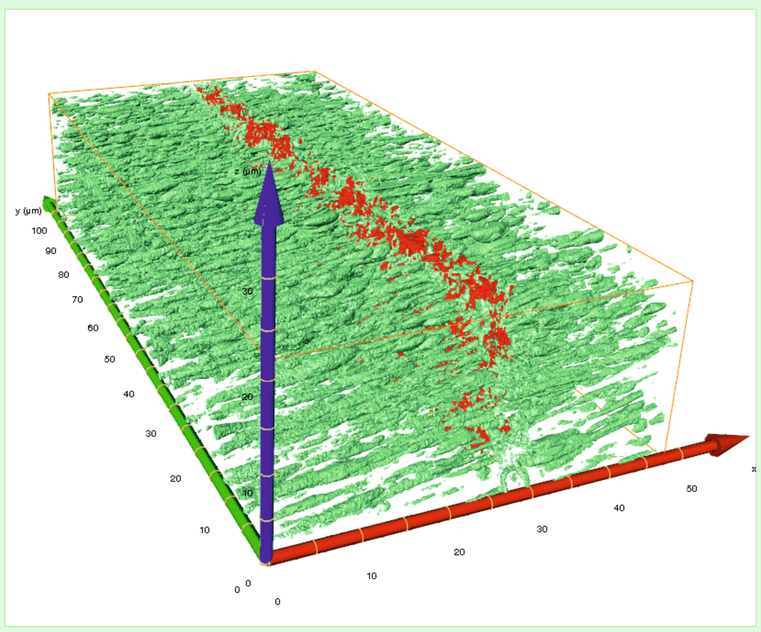
D Results and conclusion

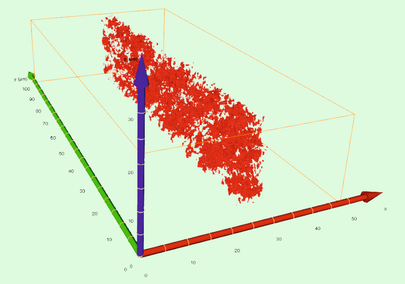
PP-EPR sample

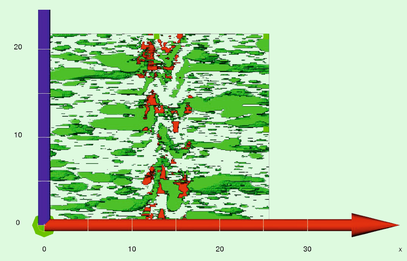


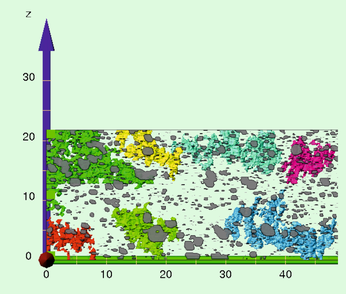
3D reconstruction of a PP-EPR sample where the tensile test was stopped at 25 % yield, with main crack (red), developed cracks (yellow) and the EPR-particles (blue).

PP-LLDPE sample









Final 3D reconstruction of the PP-LLDPE sample where the tensile test was stopped at 50 % yield, with developed voids in red and the LLDPE-particles in green.
Detailed description can be found in the text.

Examples of the obtained 3D reconstructions of the two samples are shown in the figures above. The three images on the left side show an extraction of the PP-EPR samples where the tensile test was stopped at 25 % yield and the four images on the right show parts of the LLDPE-sample where the tensile test was stopped at 50 % yield. They disclose detailed information about the microscopic fracture mechanisms in the particle modified PP. It is clearly visible that different responses to the tensile force appear for the ethylene propylene rubber (EPR) modified PP and PP modified with linear low density polyethylene (LLDPE). While widespread cracks inside the PP matrix were formed in the EPR modified samples, in case of the LLDPE modified sample voids were formed only in small bands perpendicular to the applied force, see e.g. upper LLDPE detail: although the respectively applied force was lower for the EPR sample as for the LLDPE sample. The two details of the EPR sample show, that the EPR particles are encompased by a single connected crack. Furthermore, the calculation of the EPR interparticle distance, by distance transformation, revealed that the cracks are mainly located at positions of small interparticle distances. The investigations of the LLDPE sample revealed a deformation of the particles in regions where the voids are located, see middle LLDPE detail. The lower LLDPE detail shows, that single voids (coloured) can have a bigger diameter than the diameter of the LLDPE particles (grey).

Acknowledgements and references

The authors thank the company Borealis AG (Linz, Austria) for providing materials and the ASEM for the financiel support.

[1] A. Zankel, J. Wagner, P. Pölt, (2014), Micron, vol. 62, pp. 66–78, Jul. 2014.
[2] P. Pölt, A. Zankel, M. Gahleitner, E. Ingolic, C. Grein, (2010), Polymer, vol. 51, pp. 3203-3212

Contact

www: www.felmi-zfe.at
e-mail: manfred.nachtnebel@felmi-zfe.at