Polymer fracture
3D reconstructions as an analysis tool

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Introduction

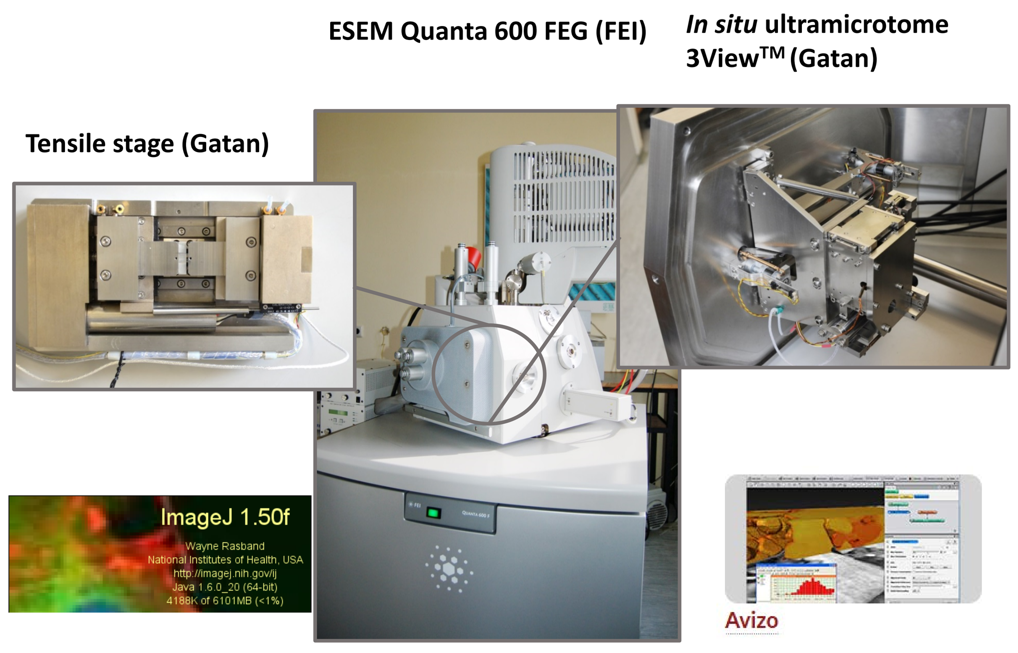
Rubber particle modified polymers are widely used materials for already a long time. Nevertheless, the fracture behaviour of such polymers is still a heavily discussed topic.

Wu claimed already in 1988 that the transition between tough and brittle behaviour of rubber blends depends mainly on the surface-to-surface interparticle distance [1].

From stress-strain curves or single 2D images of the cross sections of fractured samples, it is difficult to extract which microscopic structures / processes were mainly responsible for the fracturing and which rather tend to prevent crack growth.

But from 3D reconstructions of the fracture zone, after tensile tests stopped at forces / stresses far below the yield, it should be possible to get correlations between e.g. particle distributions, interparticle distances and crack / void formation.

Instrumentation



Experimental procedure

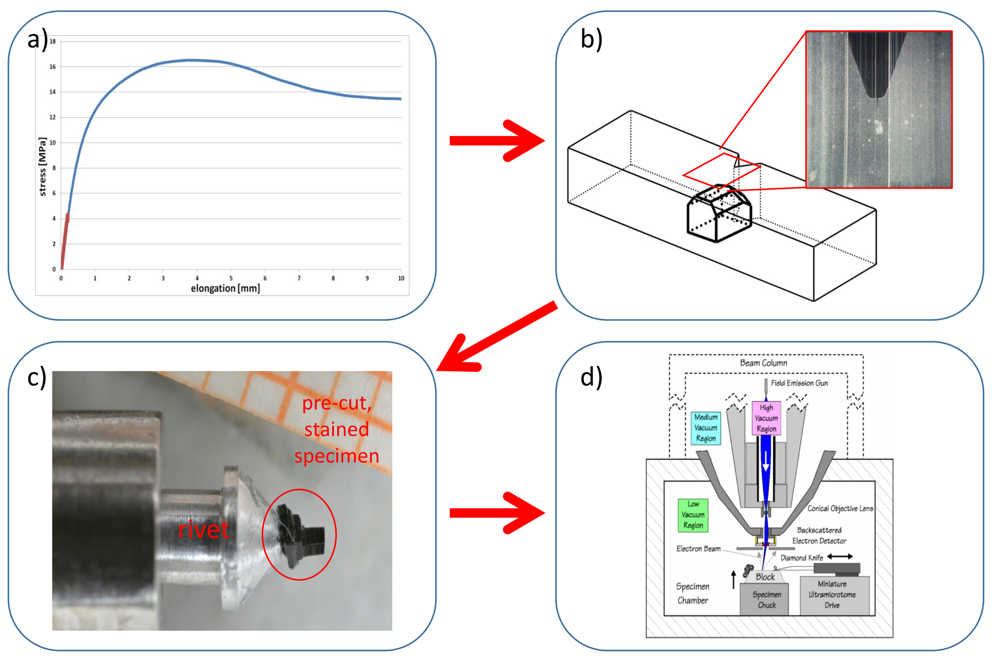


Fig. 1: Schematic representation of the experimental procedure. a) Tensile test stopped at a predefined force, far below the yield; b) part of the fracture region is extracted; c) sample embedded in resin and stained with RuO_4; d) serial sectioning and imaging. The material used was ethylene-propylene-rubber (EPR) modified isotactic polypropylene (iPP).

Surface-to-surface interparticle distance, stress concentration

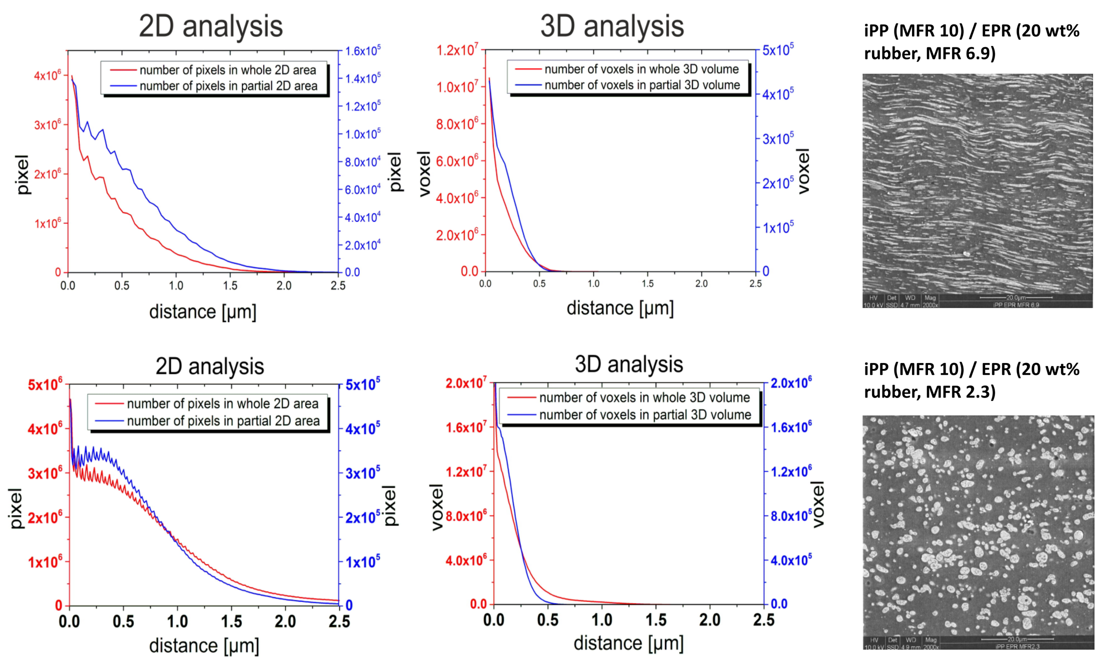


Fig. 2: Left and centre: no. of pixels (2D) and voxels (3D) in dependence on the distance from the nearest EPR particle surface in EPR modified iPP samples, determined from both 2D images and 3D reconstructions (whole volume: top: 30.0 x 31.0 x 8.5 µm³, bottom: 74 x 74 x 9 µm³; partial volumes: 7.5 x 7.5 x 7.5 µm³). Right: SEM images of cross sections of the samples.

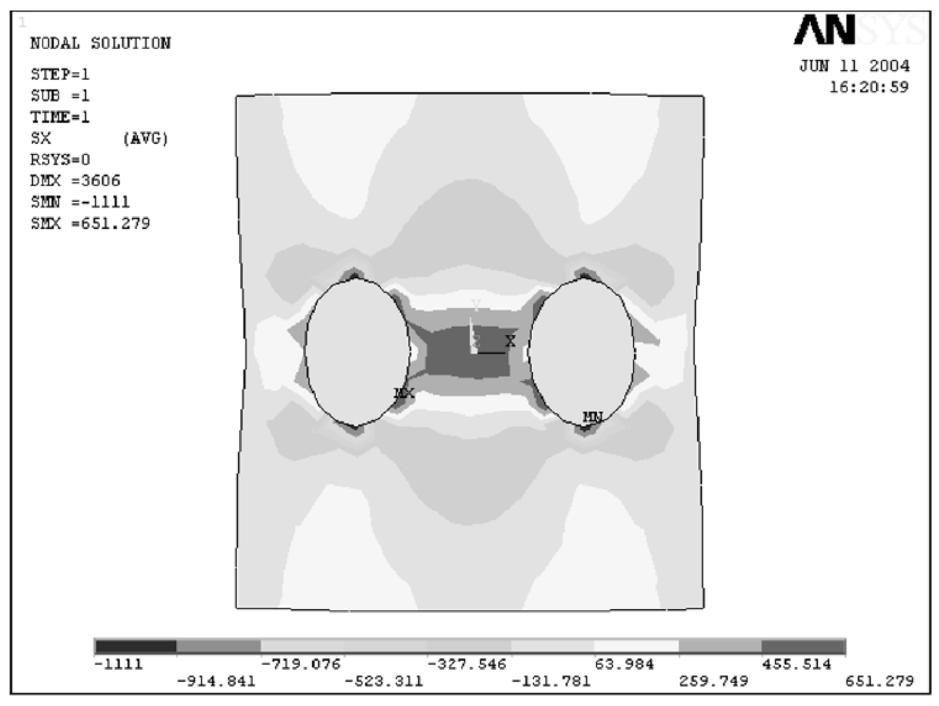
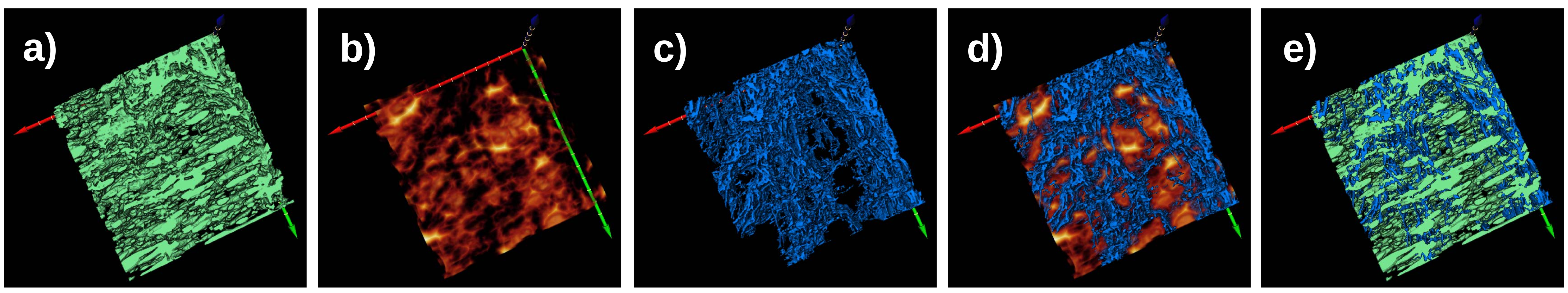


Fig. 3: Stress contour graph between two rubber particles along the X-direction in a polypropylene rubber blend (from J.Z. Liang and L. Wang [2]). The tensile load was applied in the Y-direction. The stress is maximal near the particle equators.

➔ Fig. 2 shows essentially the distribution of the surface-to-surface distances of the particles. In case the particles have varying shapes and the distribution of the particles is neither homogeneous nor isotropic, only 3D reconstructions will provide reliable distance distributions.

➔ Fig. 3 demonstrates, that strong stress concentration can happen between particles with short surface-to-surface distances. This can initiate cracking between these particles. Calculations of stress concentration between particles by G.H. Michler gave similar results [3].

3D reconstruction of ethylene-propylene-rubber (EPR) modified polypropylene (PP) after a tensile test



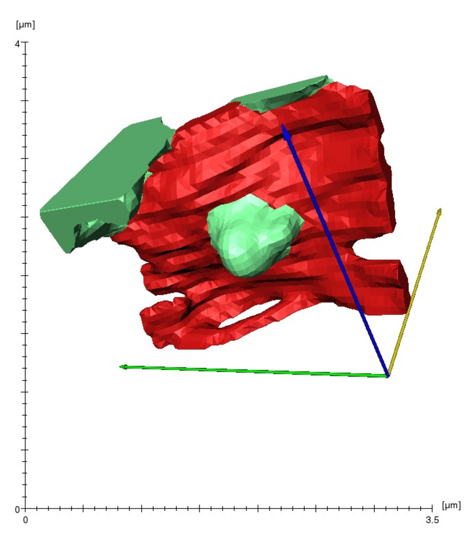


Fig. 4: 3D reconstruction of a part of the damaged region of an EPR modified PP sample, with the tensile test stopped at 50% yield (volume: 30 x 32 x 9 µm³). a) 3D reconstruction of the EPR particles; b) 3D representation of the respective Euclidean distance map (surface-to-surface) of the particles, the higher the brightness, the larger the distance between neighbouring particles; c): 3D reconstruction of the cracks; d): superposition of the distance map and the 3D reconstruction of the cracks; e): superposition of cracks and EPR particles [4].

Fig. 5: EPR particle (green) surrounded by crack (red).

➔ A comparison of the Figs. 4b) - 4d) demonstrates that cracking happens preferentially between particles with a short surface-to-surface interparticle distance. Cracking does not seem to happen between particles with a large interparticle distance. This is not so clearly visible in an overlay of the 3D reconstructions of the cracks and particles in Fig. 4e. Cracks concentrate mainly in regions where particle agglomeration appears. Fig. 5 shows that particles can be completely encircled by cracks.

➔ The 3D reconstructions enable the study of crack formation, e.g. where the very first cracks start, where they stop, how they propagate and how cracks and impact modifier resp. filler particles are connected to each other.

References

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