Load-cycling Fatigue Tests of Microfibrillar Reinforced HDPE/PA blends

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Experiment and Motivation. Time-resolved 2D SAXS measurements are repeated at HASYLAB beamline A2 during load cycling of polymer fibers with a a cross-bar speed of 0.5 mm/min of the HASYLAB tensile tester. The wavelength of the X-ray beam is 0.15 nm, and the sample-detector distance is 2542 mm. Scattering patterns are collected by a 2D detector (marccd 165; mar research, Norderstedt, Germany) operated in 1024 × 1024 pixel mode. During the deformation experiments, scattering patterns are recorded every 30 s with an exposure of 23 s. On the same materials the results of simple straining studies ("straining until failure") from 2008 have been published [1]. Now load-cycling measurements of 2008 have been repeated, because a peculiar phase shift between macroscopic and nanoscopic strain had been observed in the older measurements that had to be verified.

Summary of Results. Between November 2008 and April 2009 the control program of the HASYLAB tensile tester has been modified so that it is now possible to define the pre-straining branch and the following cycling branches in one single script. In 2008 we had to define 2 scripts and had to switch manually between pre-straining and cycling.

The phase-shift from 2008 is not verified. In several tests the behavior of the old control program is simulated and the reason for the former artificial phase shift is found: When the central control computer stops the measurement, only the recording of video snapshots is stopped immediately, whereas the recording of tensile data is continued until a new program segment is started. This behavior leads to an artificial time-offset that causes the time-shift recorded in 2008. A paper reporting first results of the new load-cycling study has been submitted [2]. Figures 1 and 2 show the evolution of macroscopic and nanoscopic parameters for cycling about a low and a high pre-strain, respectively. In the case of low pre-strain the material shows weak fatigue only, whereas

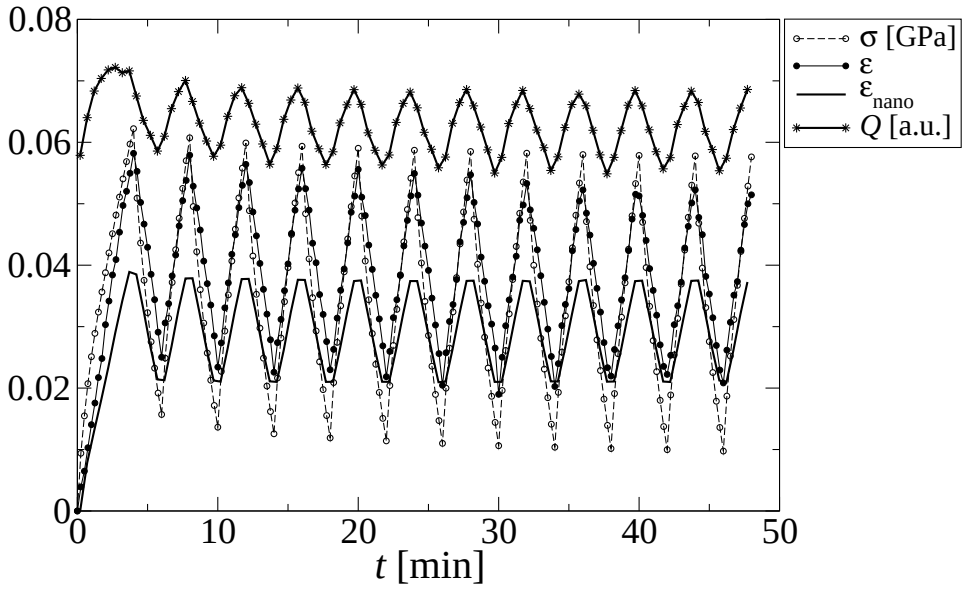

Figure 1: HDPE/PA6 (80/20) cycled about low pre-strain

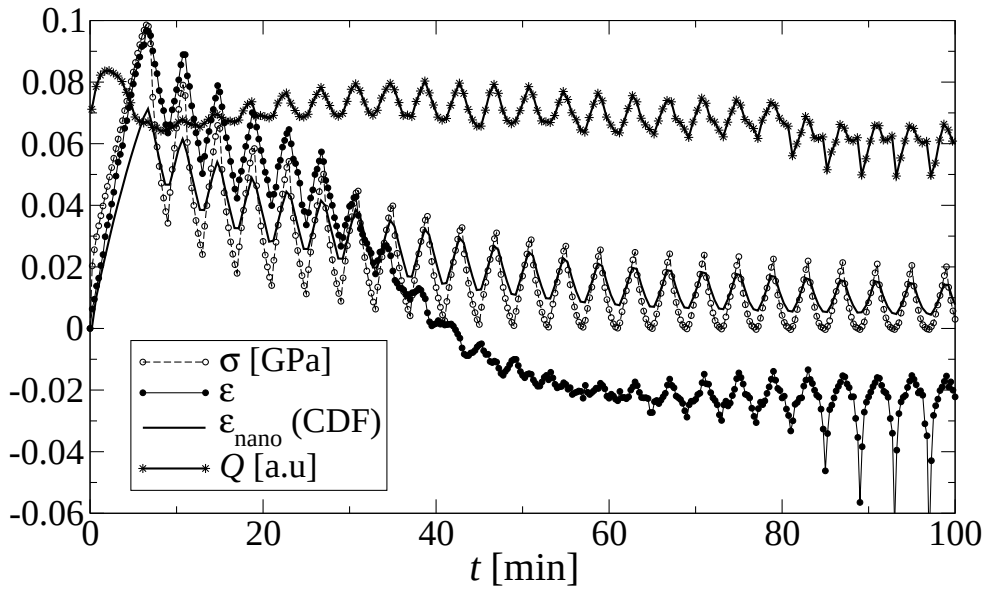

Figure 2: The material in the high-cycling experiment is showing considerable fatigue

the same material subjected to a higher pre-strain shows very strong fatigue which can be easily inferred from the decrease of the stress, σ, from cycle to cycle. In Fig. 2 the macroscopic strain, ε, becomes negative after ca. 40 min. This observation may be explained by viscous flow of the material. During the further course of the experiment ($t > 80$ min) this fatigue flow even causes the sample to become slack. It bends in each cycle close to the lower dead centers. As a result, the apparent ε drops considerably. The reason is in the method of determination. As the fiducial-mark grating is tilted with respect to the original direction of the fiber by an angle α, its lattice constant L_m appears shortened to $L_m \cos\alpha$ with respect to the orientation of the static region in the video that is evaluated to determine ε. Additionally, in this region an artificial drop of the invariant Q is observed. The reason is of geometric nature, as well. Here the bent sample has moved out of the X-ray beam. As a result, the values of $Q(t > 80\,\mathrm{min})$ cannot be discussed in terms of structure evolution. Similar is valid for $\varepsilon\,(t > 80\,\mathrm{min})$ in the high-cycling experiment (Fig. 2).

No significant phase shift is observed between ε_{nano}, σ, and ε both for low pre-strain and for high pre-strain. Thus, these quantities predominantly reflect the forced oscillation of the cross-heads. On the other hand, for both pre-strains during the first cycles the nanoscopic strain, ε_{nano}, is considerably lower than the macroscopic strain, ε. This observation shows that initially the elastic modulus of the semicrystalline stacks is higher than that of the surrounding amorphous matrix. Later, as the macroscopic relaxation starts in the high-cycling material, the nanoscopic strain of the semicrystalline stacks is unaffected and keeps cycling. This shows that the viscous flow of the material during stress relaxation is predominantly taking place in the amorphous matrix that is far away from the embedded crystallites.

References

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