In-situ SEM analysis of the tensile properties of microscale epoxy specimens

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The specific build-up of fiber reinforced polymer composites with individual fibers surrounded by matrix material, results in the presence of very small zones of matrix material (order of µm) in between the individual fibers. These micro volumes of matrix material tend to have different properties than is expected from standard testing of bulk scale specimens. This has important consequences towards the deformation behavior and especially the microscale modelling of composite materials, where the current material models are still lacking.

We performed optical and scanning electron microscopy with in situ mechanical testing to observe the deformation behavior of microscale epoxy. A big difference in deformation was observed between bulk scale epoxy and microscale epoxy samples. At the microscale level, the matrix is able to deform plastically and has the possibility to undergo large amounts of strain up to 100%. This is accompanied by a decrease in diameter of the microscale fibers, as visible in Figure 1. In contrast, at bulk scale the epoxy fractures at 8% of strain and without visible plastic deformation. Besides larger amounts of strain, a substantially higher ultimate tensile strength is observed at the microscale level.

Over one hundred microfibres with diameters ranging from 30 to 400 µm were produced. The specimens were cured conform the prescribed heating cycle of the resin supplier. This provides the possibility to compare both micro and macro (bulk) scale with each other. Tensile tests showed that the microscale behavior is clearly different, and has extreme ductility not observed in the bulk scale brittle behavior. This is accompanied by in-situ electron microscopy to unravel the deformation behavior of the micro-specimens during tensile loading. Subsequent series of deformation images enabled a detailed study of the elongation and necking process present in the microfibers when loaded. The confined microsize epoxy volumes have a plastic deformation behavior resulting in a substantially higher ultimate tensile strength (up to 380 MPa) and strain at break (up to 130 %) compared to their bulk counterpart (68 MPa and 8%). Polarized light microscopy confirmed the internal epoxy network structure rearrangement during loading.

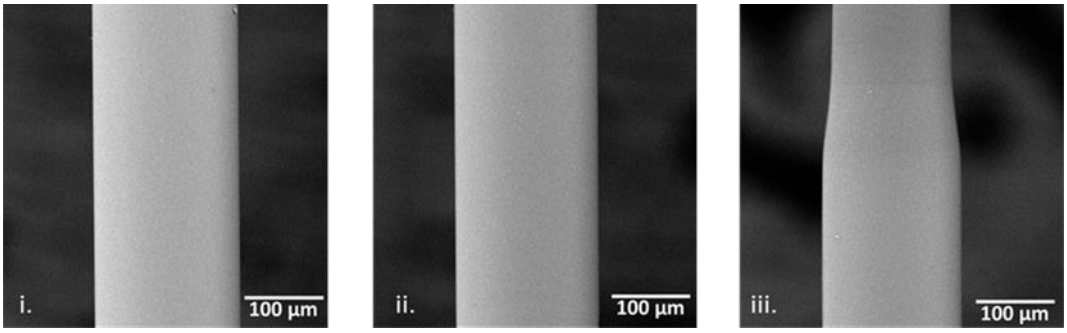

Figure 1: Plastic deformation occurring in a microfibre resulting in a large decrease of the diameter.

It can be concluded that for accurate understanding and modelling, data of the microscale behavior is inevitable. Our work provides a step forward through in-situ scanning electron microscopy analysis of this deformation behavior. This innovative technique has the potential to be used more widely, for the measurement of microscale deformations e.g. small composites, individual fibers and fabrics,…