Temperature-Controlled in-situ Tensile Tests of Polymer Tape with Single Particles

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Abstract

X-ray Computed Tomography (CT) combined with mechanical testing can deliver great insights in the damage mechanisms of polymers. Depending on the application area of the polymer, temperatures can be very high or low – leading to a different performance compared to room temperature. In this study, the influence of the temperature on damage mechanisms of a single particle in a polymer matrix should be investigated. Thus, three polymer tensile test specimens (length 30 mm; width 10 mm) – each containing a single particle – were cut out of a tape with a thickness of 0.5 mm and tested at various inspection temperatures. CT measurements were performed with the RX Solutions Easytom 160 laboratory CT device with a voxel size of (3 µm)³. Each specimen was scanned twice, namely at 0% strain and 30% strain. As inspection temperatures 0 °C, 22 °C and 60 °C were chosen. It is shown that temperature can have a great impact on the performance and damage mechanisms of a polymer, especially if a particle is present in the polymer matrix.

KEYWORDS: in-situ computed tomography; material characterization; combined loading; defect characterization

1. Introduction

Polymers are of great interest for various applications. Depending on the application field, polymers are exposed to tensile or compression forces and high or low temperatures. To predict a components failure, the understanding of damage mechanisms present in a polymer is a key enabler for component design as well as optimizing manufacturing processes. X-ray computed tomography in combination with mechanical testing allows the characterization of microstructure and damage propagation in three dimensions [1-3]. High resolution in-situ measurements have become more and more common over the years [4-7]. Several testing procedures are technically possible: (I) post mortem, (II) ex-situ, (III) interrupted in-situ and (IV) continuous in-situ [4]. In case of (I) and (II) mechanical testing is performed separately from the CT investigations. A major
disadvantage of these technique is that samples undergo long relaxation times due to de- 
and remounting which can influence the observed damage mechanism. (III) and (IV) 
require a testing rig which can be mounted into the CT device. For both, mechanical 
testing can be directly performed within the CT device. In case of (III), the tensile test is 
interrupted at certain load steps whereas this is not the case for (IV). A main requirement 
for (IV) is that the microstructure remains the same for the duration of the scan. In each 
case (I) to (IV) mechanical as well as heat treatment can be combined. To perform 
mechanical testing within the CT device various testing rigs were published by research 
groups and companies, enabling various mechanical testing methods within the CT device 
[8-10]. Also the University of Applied Sciences Upper Austria in Wels realized a 
modification of the commercially available CT500 Deben tension/compression in-situ 
stage, introducing the option for mechanical testing at selected temperatures within a 
range of –10°C up to 100°C [11]. In this study, the influence of temperature on damage 
mechanisms of a single particle in a polymer matrix was investigated.

2. Materials and Methods

2.1 Sample Preparation

Three polymer tensile test specimens – each containing a single particle – were cut out of 
a tape with a thickness of 0.5 mm. Geometry and dimensions are shown in figure 1.

![Figure 1: Illustration of the specimen geometry for tensile tests](image)

2.2 in-situ Tensile Testing

For temperature-controlled in-situ tensile testing the MIST (Mechanical In-situ Stage 
with Temperature control) – a modification of the commercially available CT500 Deben 
tension/compression in-situ stage – was used, which was developed at the University of 
Applied Sciences Upper Austria in Wels [11]. For these experimental series 0 °C, 22 °C 
and 60 °C were chosen as inspection temperatures. For the inspection temperature 0 °C 
and 60 °C, the testing procedure was as follows: (I) hold at 22 °C, (II) heating or cooling 
until inspection temperature is reached, (III) tensile test at inspection temperature and 
(IV) cooling or heating until 22 °C. In case of 22 °C as inspection temperature only step 
(I) and (III) were performed. Tensile tests were performed until a strain of 30% was 
reached with an elongation speed of 0.5 mm/min. Temperature profile was recorded with 
the MIST control software [11], force profile was recorded with the Deben Microtest 
software V6.1.83 © Deben UK LTD.
2.2 Computed Tomography and Data Evaluation

Computed tomography measurements were performed with the RX Solutions Easytom 160 (RX Solutions) equipped with a 160 kV Hamamatsu Nano focus tube, a LaB6 filament, and a 1920 pixel × 1536 pixel Varian flat panel detector. A voxel size of \((3 \mu m)^3\) was achieved with this setup. The following scan parameters were used: a voltage of 80 kV, a current of 60 mA, an integration time of 500 ms and 2160 projections. Each specimen was scanned twice, at (I) 0% strain and 22 °C and at (II) 30% strain and inspection temperature. Scan time was 90 minutes per scan. Volume data was reconstructed using the filtered back projection algorithm of the xact64 reconstruction software (RX Solutions). Subsequent image processing steps and data evaluation were performed in VGStudio Max 3.5.2 (Volume Graphics).

3. Results and Discussion

The particles present in the polymer specimens were segmented and 3D renderings at 0% strain and 30% strain are shown in figure 2.

![Figure 2: Segmented particle present in the polymer test specimens. The following pairs show the same particle: (a) and (d), (b) and (e), (c) and (f). (a) – (c) show the particles before tensile testing at 0% strain and 22 °C; (d) – (f) show the particles after tensile testing at 30% strain and inspection temperatures: (d) 22 °C, (e) 60 °C and (f) 0 °C. Formed defects are illustrated in green and blue ((d) and (f)).](image)

Regarding the tensile tests at 22 °C (figure 2 (a) and (d)) and 0 °C (figure 2 (c) and (f)) both polymer test specimens showed defect formation. In case of the tensile test at 22 °C...
the particle cracked three times, leading to rupture of the particle into smaller parts. Between these parts defects were formed. In comparison the tensile test at 0 °C shows, that the particle did not crack but instead defects formed above and below the particle. This can be due to different materials of the particle (and therefore differences in adhesion between polymer matrix and particle) or due to their difference in geometry or even both. Whereas, the particle of the tensile test at 0 °C shows similar dimensions in x-, y- and z-direction, the particle of the polymer specimen which was tested at 22 °C is very thin in x-direction compared to y- and z-direction. In case of the tensile test at 60 °C (figure 2 (b) and (e)), no defects were formed. Instead, an elongation of the particle in z-direction was observed. This is probably due to the fact, that at elevated temperatures polymers can get softer and consequently can be deformed more easily. Further, the forces recorded were significantly lower compared to the tensile tests at 0 °C or 22 °C with only 16 N compared to 80 N (0 °C) and 100 N (22 °C) which is also indicating that the polymer matrix is way easier deformed when exposed to a temperature of 60 °C.

4. Conclusion

In summary, mechanical testing of a polymer tape with single particles was performed at three different temperatures. Whereas the measurements at 0 °C and 22 °C both showed defect formation and similar recorded forces, the sample tested at 60 °C behaved differently concerning defect formation and stress-strain behavior. In case of the high temperature test, no defects were formed. Instead, the particle did elongate in z-direction with the deforming polymer matrix. In conclusion, application temperature can have a great impact on the performance of a polymer. Thus, making mechanical tests that combine the application of force and temperature at the same time crucial for understanding potential damage mechanisms at certain application temperatures.

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References


