Quantification of the Resistance against Deformation under Thermal Load of Different Polymeric Foams by a Novel Measuring Approach

Tobias Standau, Andreas Himmelsbach, Catharina Stierle, Johannes Meuchelböck, Volker Altstädt and Holger Ruckdäschel[a)]

University of Bayreuth, Department of Polymer Engineering, Universitätsstr. 30, 95447 Bayreuth, Germany

[a)] *Corresponding author: holger.ruckdaeschel@uni-bayreuth.de*

Abstract. In recent years, many (bead) foams have been developed that are claimed to have an elevated resistance to thermal stress. However, there is no uniform technique established to quantify this property; instead, numerous methods exist that vary in quality and reproducibility. When they are all compared, two drawbacks can be identified: (i) these tests usually apply a temperature ramp, which, due to the thermal inertia of foams, leads to temperature gradients within the sample and thus to less reliable results, and (ii) a commonly applied (fixed, constant) mechanical load impairs the possibility of comparing different foams (e.g., different materials, structures, and/or densities). Therefore, from a technical point of view, we have developed a novel approach by combining a static compression test to determine a (relative) test load for each individual foam, which is then applied in a steady creep test with defined temperature steps. Yet, it is possible to quantify a temperature for resistance to thermal deformation (under compression); we propose to call this temperature "heat stability temperature T_{HS}". We have applied this test to several foams with different densities and foam structures. For example, we were able to show that EPET exhibits a higher temperature resistance than EPP. Furthermore, the T_{HS} for foams follows the same trend as the heat deflection temperature HDT, obtained from compact samples.

INTRODUCTION

Much has happened in the field of bead foams in the last two decades [1]. Besides sustainability (e.g., bead foams made from PLA) and enhanced mechanical properties (e.g., bead foams made from TPU) one trend is the establishment of bead foams made from engineering polymers such as PET [2] or PBT [3, 4] with increased service temperatures. It has often been described that the melt strength - especially of polyesters - is rather low and the expandability is essentially hindered. A typical strategy is the addition of chemical modifiers during extrusion [5, 6].

However, a fundamental issue is the quantification of the increased thermal stability of the foams. It is well known that mechanical properties (e.g., the compression behavior) depend on temperature [7]. A variety of techniques express the withstand against thermal stress in either qualitative or quantitative ways exist, but their results are not comparable, especially when foams with different densities are studied. Two major drawbacks can be identified. First, when a temperature ramp is applied to an insulator such as a foam, temperature gradients occur within the sample, which distort the results. Second, a constant load is commonly applied in these tests, that will have a very different impact solely depending on the foam's density and -structure, respectively. For instance it is known that small cell sizes can result in higher compression loads [8]. Moreover, bead foams exhibit a kind of superstructure consisting of the beads themselves and voids in between (macro-level) and cells within these beads (micro-level), where the deformation is strongly governed by any inhomogeneity that occurs, as shown by Ossa et al. [9]. Therefore, we have developed a

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new two-step approach [10], where (i) a very low load is determined from a compression test at room temperature, which is then (ii) applied in a creep test with defined temperature steps until a relative deformation of 10% is reached. Here the temperature steps are adjusted to achieve equilibrium between the heating chamber and the specimen core. The approach is now applied to bead foams from PET aiming to compare them with other bead foams (i.e., EPS, EPP, E-PBT).

EXPERIMENTAL

Compression Tests at Different Temperatures

Compression tests were performed on E-PBT cuboids (40 x 40 x 20 mm) with a density of 229 kg/m^3 at different temperatures to determine the temperature dependence of the compression stress and the Young's modulus. In our previous work, the composition and processing of this material was described in detail [4]. The tests were carried out on a Zwick Roell universal testing machine with a heating chamber with a test speed of 2 mm/min and a preload of 10 N. The selected ambient temperatures were 25, 50, 75, 100, 150 and 200 °C, respectively.

Approach to determine the Heat Stability Temperature T_{HS}

The approach used in this study has been described in detail in a previous paper [10] and can be seen in Figure 1. First, a static compression test is performed at room temperature on a universal testing machine (Zwick Roell) to determine a very small load, which is later used as the test load in a steady creep test with temperature steps (ΔT=10 K) on a servohydraulic testing machine (Instron). The test criterion is 10 % relative deformation of a cuboid specimen (40 x 40 x 20 mm). Two different grades of E- PET with different densities from Armacell were used. Furthermore, materials known from the previous study were also used for comparison.

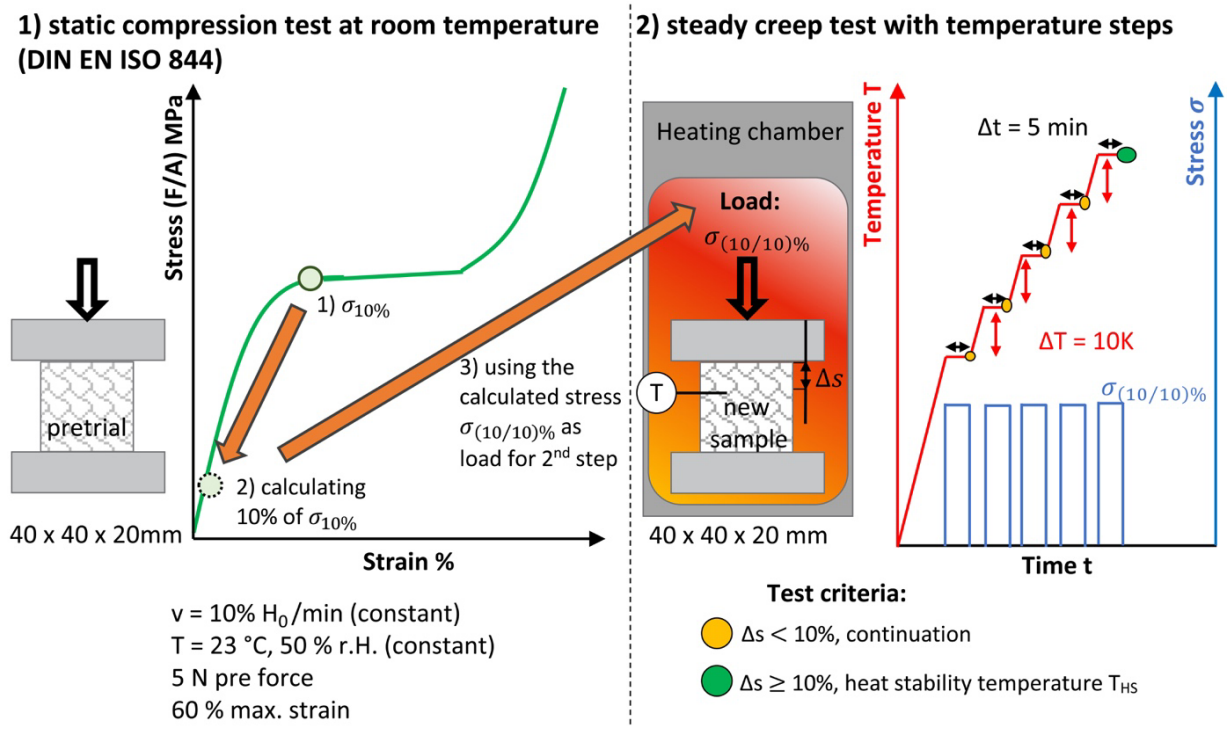

FIGURE 1: Scheme of the measurement principle. In a first step (left) compression test according to DIN EN ISO 844 are carried out to determine a very low load ($\boldsymbol{\sigma_{(10/10)\%}}$), which is applied in the second step (right) – a creep test with temperature steps. (image by Himmelsbach et al. [10], used under CC BY 4.0)

FIGURE 2: Results of compression tests carried out on E-PBT (229 kg/m^3) at different temperatures.

Figure 2 shows the stress-strain curves of E-PBT in compression at different temperatures. Less force is necessary to deform the specimen at more elevated temperatures.

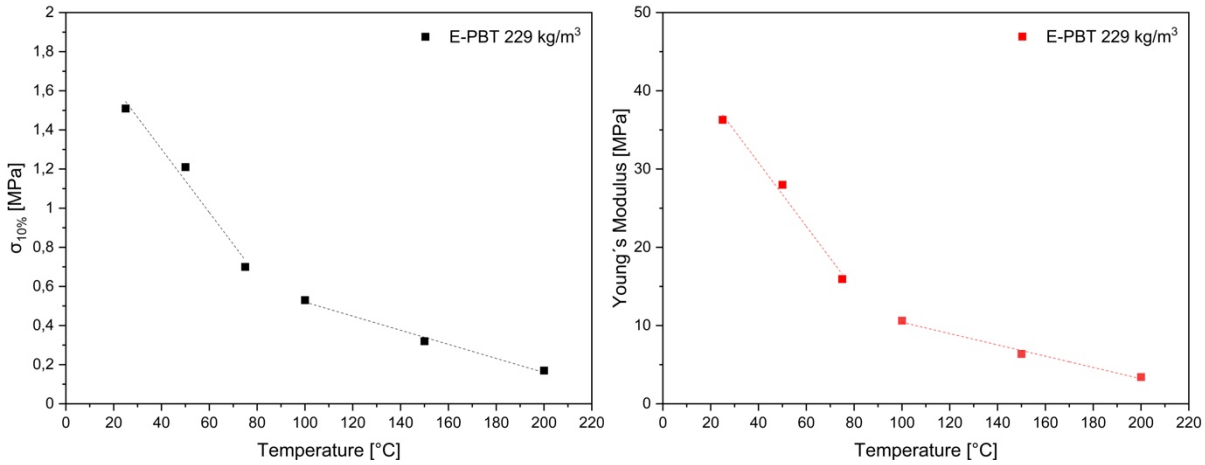

FIGURE 3: Temperature dependency of the stress at strain (10%) and the Young´s modulus of E-PBT taken from the compression tests.

Figures 2 and 3 show that E-PBT resists lower compressive stress at a higher temperature. It is obvious that both stress and the Young's modulus decrease with increasing temperature. Similar results were found by Morton et al. for EPP in the range between -30 and 60 °C [7]. Interestingly, this decrease occurs in two phases, and the slope appears to be linear in both cases. Up to T_g, the decrease in strain (and modulus) is much steeper and flattens out when it exceeds this value. Below T_g, the foam is much stiffer, and more compressive stress must be applied to deform the specimen. Above the T_g, the polymer chains gain mobility, and deformation requires much less compressive stress. Here, creep is the main cause for deformation of the specimen.

Determination of the Heat Stability Temperature T_{HS}

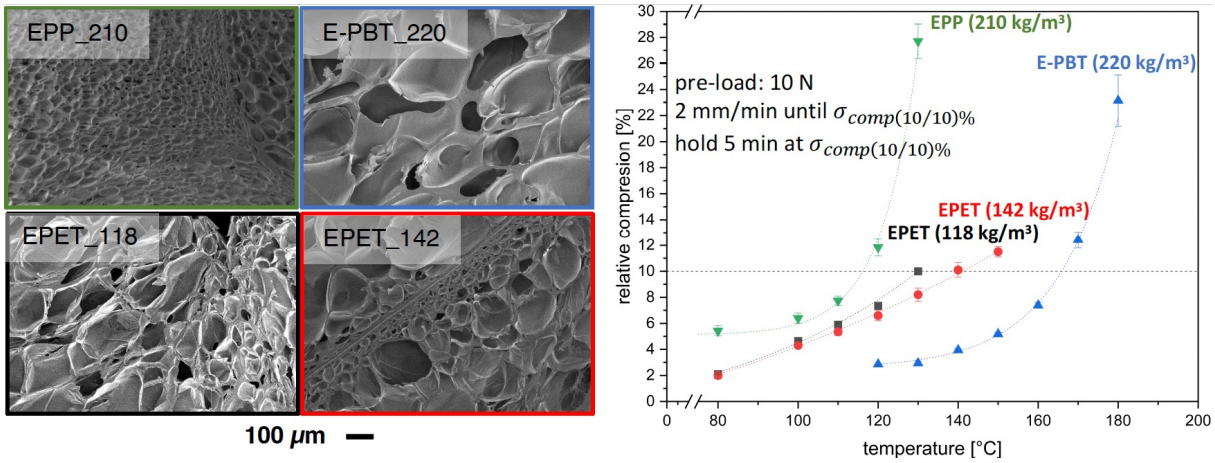

FIGURE 4: Left: SEM images of the investigated foam structures. Right: Relative compression during the creep test at different temperature steps

In Figure 4 the foam structures are exhibited and the relative compression over the temperature obtained from the creep tests with temperature steps is shown for EPET (118 and 142 kg/m^3, respectively), E-PBT (220 kg/m^3) and EPP (210 kg/m^3). The test criterion of 10 % relative compression for the heat stability temperature T_{HS} was achieved at 130 °C (EPET_118) and at 138 °C (EPET_142), respectively. The slight differences for EPET could be due to the fact, that the samples were prepared from two different grades.

Comparing the newly measured data for EPET with the existing data for EPS, EPP, and E-PBT from our earlier article [10], it becomes clear that the engineering polymers can withstand the thermal stress in this creep test (2nd step) much better than the established bead foam materials polystyrene and polypropylene resulting in much higher T_{HS}.

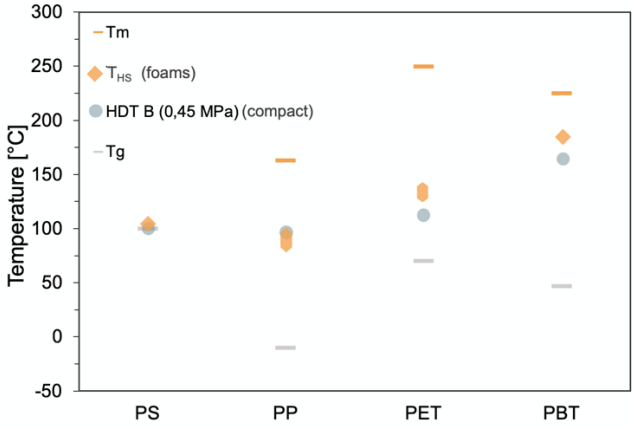

FIGURE 5: Thermal data (T_g and T_m) as well as heat deflection temperature (HDT/0.45 MPa) extracted from data sheets as well as the T_{HS} measured with the described approach (data partially taken from Himmelsbach et al. [10]).

Figure 5 summarizes the thermal data (T_g and T_m) of various thermoplastics used for bead foams. Furthermore, from data sheets the heat deflection temperature HDT/0.45 MPa is given. While for amorphous polymers it can be expected around the T_g, for semi crystalline thermoplastics it is typically located between T_g and T_m. Interestingly, the HDT for PET is lower than for PBT, although PET has a higher T_m. The same trend is observed when looking at T_{HS}. A plausible reason for this could be the different crystallization behavior of these polymers. PBT crystallizes much

faster and to a higher extent. It is well known that crystallinity is a key factor for thermal stability, as exemplified by foamed PLA sheets [11].

CONCLUSION

In this study, the behavior of different foams under thermal stress was investigated. It was shown that the Young's modulus of PBT bead foams decreases with temperature. When T_g is exceeded, the decrease in modulus seems to be more dominated by creep. The heat stability temperature T_{HS} for PET bead foams with different densities was determined according to the two-step approach. It ranges from 130 to 138 °C. Thus, it is higher than for the established bead foams made of polypropylene (i.e., EPP). Comparing the heat stability temperature T_{HS} (foams) with the heat deflection temperature HDT/0.45 MPa (compact), it can be seen that both follow the same trend. Here, PBT shows a higher resistance to deformation under thermal load than PET, which is most likely due to the more pronounced crystallinity.

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