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Experimental characterization of thermoplastics for use in heat exchangers †

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Abstract: For the prediction of long term behavior several methods are known. This paper focuses on creep in dynamic mechanical analysis (DMA) and in a tensile setup. The investigated material was Polyamide 6 (PA6). As a pre-study for the DMA, Polypropylene (PP) was tested considering five different factors. To determine the significant influences, the results were interpreted statistically.

Keywords: DMA, Creep, Polyamide 6

1. Introduction

Creep tests, the deformation of material due to permanent loading, require lengthy and therewith costly experiments. Since the discovery of the time-temperature superposition principle, a shortened testing method for rheological simple material has been found [1]. But advise is barely given for the whole process chain containing correct sample preparation, measurement of true results and application of the principle including validation with other methods. One measurement method for creep test and in general to characterize polymers, is the dynamic mechanical analysis (DMA). Under defined thermal conditions the sample is loaded with a defined force. The caused displacement is measured and the creep compliance calculated. Other possible measurements in DMA are frequency, temperature or strain sweeps to characterize the purely elastic behaving region of the sample. All measurements can be performed in different kinds of clamps: Tensile, cantilever, shear, compression or three-point bending. Information on the suitable type of clamps are provided in the machine description [2].

Typically sample material is tested at the later usage temperature. This temperature can be taken as reference temperature for the TTSP. The principle is based on the idea that time and temperature have the same effect on thermo-rheological simple material. Since long-term tests are costly, several shorter test can be performed at different temperatures instead. From the gained results a so called master curve can be generated by shifting the single curves horizontally. Under certain conditions the method can also be applied to rheological more complex material by shifting in two dimensions [3]. Generating several curves at different temperatures is less time-consuming than real-time creep tests, but for a first material screening still extensive. With the stepped isothermal method it is possible to merge several temperatures in one test. According to the testing standards only one repetition is necessary [4]. Due to the extreme reduction of testing time from several years to a test lasting a few hours, the prediction of long-term behavior is likely to fail if the principles are not applicable on the material or the test was not performed precisely. This paper should give an idea how method evaluation can be performed by using statistical analysis.

2. Materials and Methods

The experimental part is divided in two sections. The first part contains the test setup with a critical examination of the repeatability of results with special focus on the execution of a DMA test. The second part presents the long-term behavior of polyamide 6. The data will be gained with creep tests in a larger dog bone test and a smaller DMA test.

The preparation of DMA samples needs to be done precise and thoughtful to prohibit wrong measurement. The accuracy of shape is requested to be less than ± 2 % in width and thickness, but the sample size is rather vague. For the size of the sample some standards only demand a rectangular shape [5,6], ASTM D6992 provides limits: length 21-60 mm, width 2-13 mm and thickness 0.5-2.5mm [4]. Gabbott [7] suggested different thicknesses for each clamping type. Other authors recommend a span-to-thickness-ratio of greater than 10 [2,8], ASTM D790 fixes the ratio to 16±1 [9]. The sample size is also restricted by the machine-related limits of stiffness between 100-10,000,000 N/m. But even if these conditions are complied, the test result of loss modulus (E′) and storage modulus (E′′) can vary significantly (Figure 1). Due to these unpredictable fluctuation it was decided to perform a systematic study on the influences on DMA.

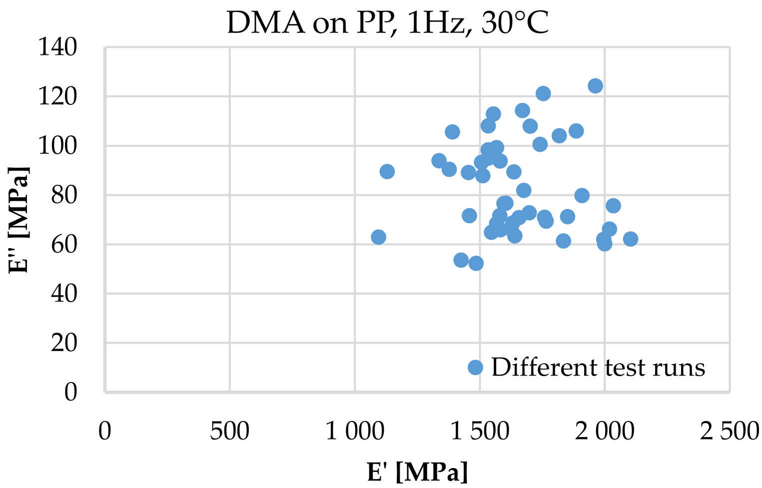

Figure 1: Variation of DMA test results despite all PP, 1 Hz, 30 °C, span to thickness ratio >10, but different clamps, production methods, dimensions and isothermal step length

The chosen trial material for the DMA in Texas Instruments' Q800 was Polypropylene (PP). As possibly relevant influences on the testing result five factors were considered:

- Clamp type
- Production method
- Span to thickness ratio (ST-Ratio)
- Width
- Isothermal step

Even if stated in literature tensile clamps are only recommended for films and fibers [1,2], we would like to perform tests in that clamp as well, because the direction of loading is the same as our tensile creep test on dog bones. As second clamp single cantilever was chosen. For comparison selected production methods were extrusion and injection molding. For the span to thickness ratio a span of greater than 10 is recommended. For the trials ratios of 10 and 16 were selected to validate if a ratio of 10 is sufficient.

Since the length in the single cantilever clamp is fixed, the thickness needed to be adapted. The extruded material could directly be produced in the required thicknesses of 1.0 mm and 1.8 mm. Injected bars were grinded down from 4 mm to the corresponding extruded thicknesses. The width was restricted by the clamps to maximum 10 mm. For the trials samples were cut to a width of 5 mm and 8 mm. Last considered influence is the length of the isothermal step; 3 min and 10 min were compared.

For the creep testing, Polyamide 6 was produced in injection molding. The material was chosen due to a higher glass transition temperature and therewith better expectable performance in the later application of a heat exchanger. For the DMA testing, bars are grinded down to a thickness of 1,7 mm and a width of 4 mm. Thereby water cooling was used to keep the surface temperature as low as possible. The rough surfaces were polished with a grain size up to 5 µm. For the creep test in the tensile machine, injected DIN EN ISO 527-4 [10] dog bones were used.

2.1 DMA

Setting up the DMA occurred according to the instructions of the machine manual and additional literature. The machine specific clamp calibration was performed at least once per day. The measurement of the length, especially in the single cantilever, was done manually after the setup was heated, because the middle clamp can move with slight pressure about 1 mm [8]. Since the length is equal to stiffness multiplied by the geometry factor and in case of the cantilever the length is cubic in the formula [2], a small error in measurements changed the result of the modulus noticeable.

For the pre-study a single frequency sweep at 1 Hz and 30 °C was chosen. The strain was 0.05 % in favor of staying in the pure viscoelastic region. In case of the tensile test a pre-stress of 0,01 N was applied to prevent buckling. According to statistics a half fractional factorial 2^5-design with three repetitions per test was performed. With the resulting value an univariate test with second order interactions was performed to find the significant factors influencing the DMA result, namely the tan δ, which was calculated from the loss and storage modulus [11].

2.2 Tensile machine

The creep test on dog bones was performed in a common tensile machine (Instron R5800) with heat chamber. Instead of using the load control setting, which leads to an overshooting of the desired load, a constant load was placed on the cross head and fixed on the sample. By moving the crosshead in a known speed, the loading phase has been defined and ends when the sample was fully loaded. Strain measurement was performed with 3D-DIC through the glass of the heat exchanger. With comparing the strain to an extensometer at room temperature it could be ensured the glass has no falsifying distortion effect, see Figure 2.

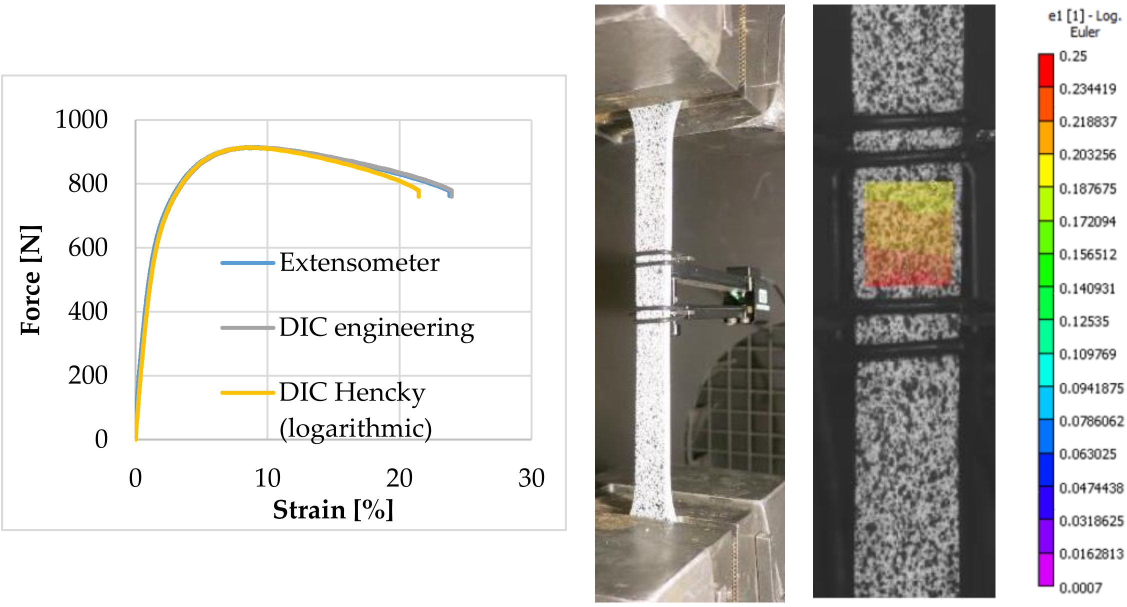

Figure 2: Comparison of DIC and extensometer measurement, good result until F_{max}

3. Results

The normal distributed results indicate, DMA testing is sensitive to many influences. Width a confidence interval of 95 %, all main factors are considered as significant except for the isothermal step. Especially the choice of the clamps cause a strong variation of the results as displayed in the boxplot Figure 3. Notable intersection is evidenced in Figure 4 and occurs according to the F-value between the production method and the span-to-thickness-ratio.

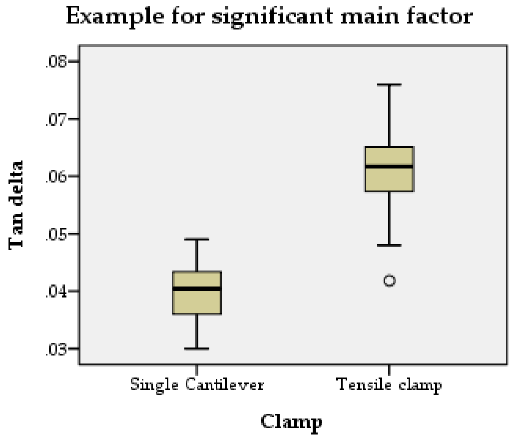

Figure 3: Boxplot diagram showing the influence of the clamp type on the resulting tan delta in DMA

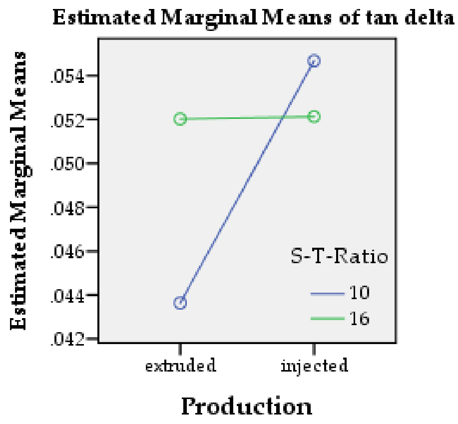

Figure 4: Profile plot picturing the intersection between production method and span-to-thickness ratio

Even if the issue of influencing factors is not entirely solved yet, a DMA creep test was performed on PA6 as an example. A loading of 1 h with subsequent 2 h relaxation was performed on a load level of 0.01 %. Also tensile creep tests were performed to generate a master curve through a curve shift. The result is shown in Figure 6.

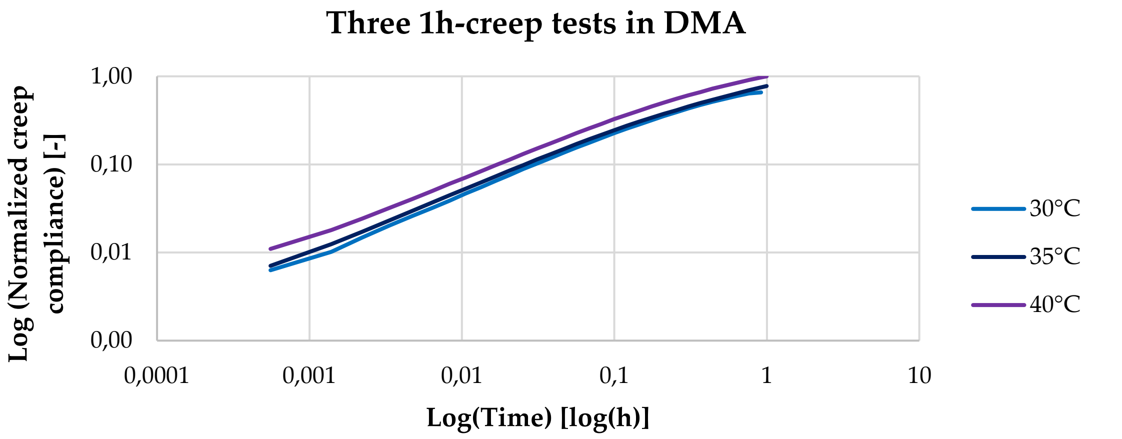

Figure 5: Creep curve of PA6, performed from 23 °C until 45 °C in 5 °C steps created from DMA test with a load according to 0.1 % of the tensile strain at a strain rate of $10^{-5}s^{-1}$

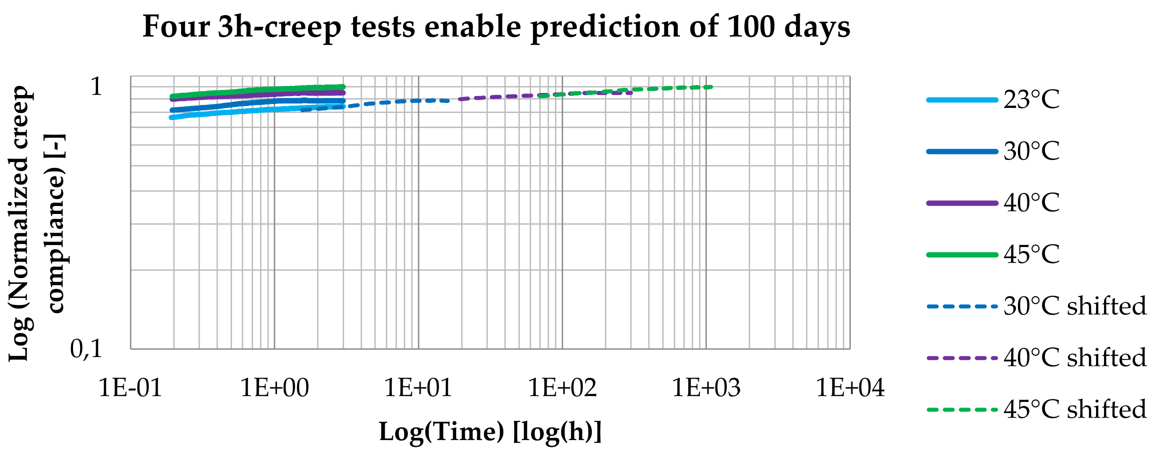

Figure 6: Master curve for PA6 created from tensile curves according to a load of 1 % of the tensile strain at a strain rate of $10^{-5}s^{-1}$

4. Discussion

Due to the strong scattering of the results, no clear trend could be detected. The clamp type had a significant influence because of the different type of loading of the sample. It was expected to notice an interaction between the dimensions of the sample and the clamp type which did not occur. Maybe the differences of width or ST-Ratio were too small to spot a significant interaction. A higher ratio or a smaller width in the tensile clamp might enable the usage.

The significant influence of the production method is unlikely caused by the production itself since the polymer grade is the same. Supposable, the post processing has an influence due to eventual different surface roughness, edges or thermal influence due to grinding.

Only the length of the isothermal step seems to have no influence on the result. The heating speed of 2 °C/min and the subsequent isothermal step of 3 min respectively 10 min seem to be enough to heat the sample through. It is supposed that the length of 3 min is long enough to heat the sample entirely through. For time saving purposes, a shorter time step or faster heating could be tried.

Due to the missing of a clear trend, it is to be assumed that important factors were disregarded until now. Another potential source of error might be for example the clamping angle. With horizontal laser lines the procedure was tried to be improved, but also due to the fragile clamp, torsion is not entirely excludable.

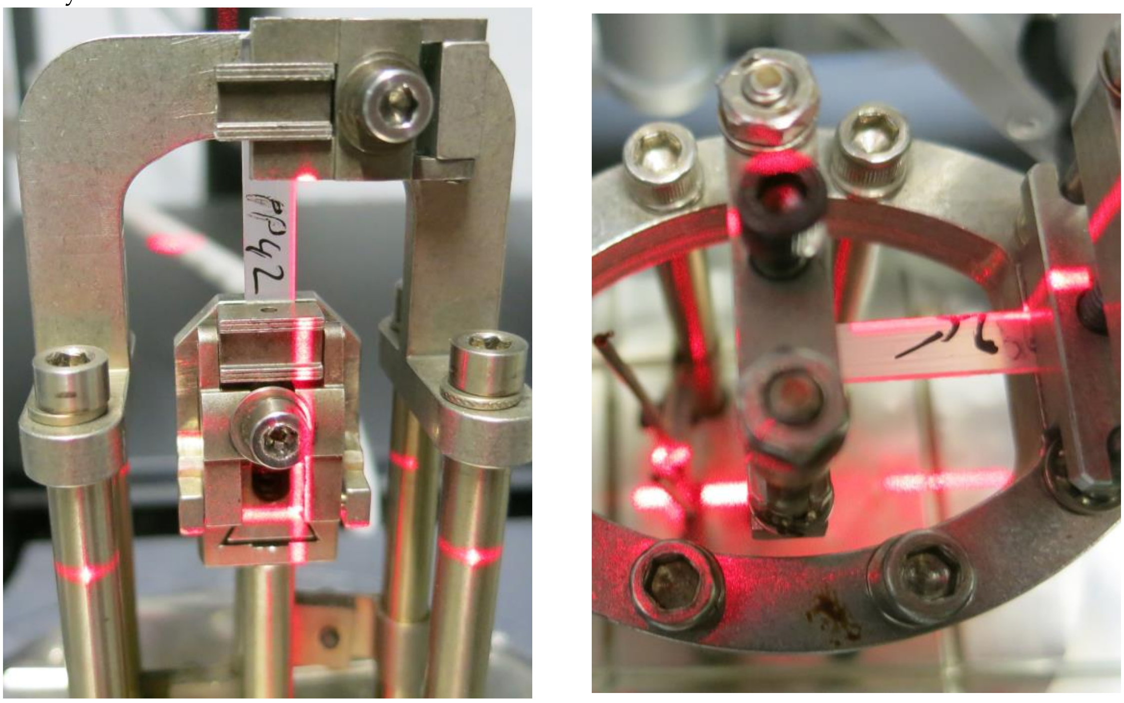

Figure 7: Aligned laser lines for straighter sample mounting in tensile clamp and single cantilever

With further investigation it will be hopefully possible to find a general method to be certain about the correctness of the gained results. Afterwards creep curves like the one showed in Figure 5 can be monitored. In combination with the shifted creep curves from the tensile setup, it should then be possible to generate load dependent long-term predictions for the material.

5. Conclusion

Influences on the procedure of dynamic mechanical analysis were examined. A statistic approach showed a heating speed of 2 °C/min plus an isothermal step of 3 min is sufficient to heat the sample core. Other results from the statistical analysis are, DMA is highly sensitive to several parameter such as clamp type, sample dimensions and preparation method of the sample. A solution to overcome the instability in measurements has not been found, yet. After solving those issues, it should be possible to compare creep tests in DMA with creep tests from the tensile machine. With the usage of the time temperature superposition principle, the performance of PA6 can be predicted for at least 100 days by only performing 12 h of testing. Further investigations will follow.

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Author Contributions: Wim Van Paepegem, Anouar Krairi and Joanna Schalnat conceived and designed the experiments; Joanna Schalnat performed the experiments and analyzed the data; Tom Wieme contributed materials, Wim Van Paepegem analysis tools; Joanna Schalnat wrote the paper.

Conflicts of Interest: The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

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