Static and dynamic thermo mechanical characterization of a bio-compatible Shape Memory Polymer

Pauline Butaud[1]*, Anne Maynadier[2], Vincent Placet[1], Morvan Ouisse[1], Emmanuel Foltête[1], Cécile Rogueda-Berriet[1]

[1] FEMTO-ST Applied Mechanics Department, Besançon, FRANCE
[2] LaMCoS Laboratoire de Mécanique des Contacts et des Structures, Lyon, FRANCE

*pauline.butaud@femto-st.fr

ABSTRACT:

The presented study deals with the synthesis and the thermo-mechanical characterization of a thermally-actuated shape-memory polymer (SMP). The chosen polymer is a chemically cross-linked thermoset. It is synthesized via photo polymerization (UV curing) of the monomer tert-butyl acrylate (tBA) with the crosslinking agent poly (ethylene glycol) dimethacrylate (PEGDMA). The mechanical characterization has been performed thanks to three kinds of tests. A quasi-static tensile test allows the identification of the Young's modulus and the Poisson ratio at ambient temperature. The DMA is used to determine the evolution of viscoelastic properties versus temperature and frequency under harmonic loading. A modal analysis has been performed to identify these properties at higher frequencies. The main purpose of this work is to check the validity of the time-temperature equivalence obtained from the DMA measurements. The predicted viscoelastic properties, illustrated through the use of master curves, are compared to the measurements collected using quasi-static and modal test.

KEYWORDS:

SMP, synthesis, characterization, DMA, modal

INTRODUCTION

Shape memory polymers (SMPs) belong to the class of smart materials. The SMPs have the ability of changing their shape in response to an external stimulus, most typically thermal activation. When the SMP is heated above the glass transition temperature Tg, it is soft and rubbery and it is easy to change its shape. If the SMP is subsequently cooled below Tg, it retains the given shape. When heated again above Tg, the material autonomously returns to its original permanent shape [1]. The increasing use of SMP for quasi-static and dynamic applications, under various temperature ranges, has made necessary the characterization of these materials over wide frequency bands [2].

MATERIALS AND EXPERIMENTAL METHODS

As a representative thermally-actuated SMP, a chemically-crosslinked thermoset polymer recently studied by Yakacki et al. [3] is chosen, the tBA/PEGDMA. The SMP is synthesized by manually mixing 95 wt% of the monomer tBA with 5 wt% of the crosslinking agent PEGDMA. The photoinitiator DMPA, is added to the solution at a concentration of 0.5 wt% of the total weight. The polymerization is initiated by exposing the solution to UV light.

The mechanical characterization of the tBA/PEGDMA has been performed thanks to three kinds of tests. A quasi-static tensile test, performed at ambient temperature, allows the identification of the Poisson's ratio using digital image correlation. Loading-unloading tests were carried out between -15 °C and 30 °C to determine the Young's modulus at different temperatures. Viscoelastic properties (storage modulus, loss modulus and loss factor) are measured using a Bose Electroforce 3200 apparatus every 5°C between -28°C and 30°C, the frequency of the solicitation varies from 0.1 Hz to 10 Hz. Moreover infrared thermography was performed during quasi-static tests and DMA to visualize

the possible overheating in the sample. Finally a modal analysis on a tBA/PEGDMA rectangular plate is performed; the response is measured by using a laser vibrometer. A model-test correlation is achieved on AESOP software and a model updating procedure is conducted to obtain the mechanicals parameters of the tBA/PEGDMA at the modal frequencies.

RESULTS AND DISCUSSION

This study intends to compare the master curves obtained with the DMA and the results collected by the modal analysis and the quasi-static tests. Based on the time-temperature equivalence determined by DMA [5], the storage modulus predicted by this equivalence should be equal with the quasi-static and modal modulus, likewise for the loss factor. The DMA master curves and the results of quasi-static tests and modal analysis in temperature beforehand translated thanks to the shift factor are plotted on a same graphic (Figure 1).

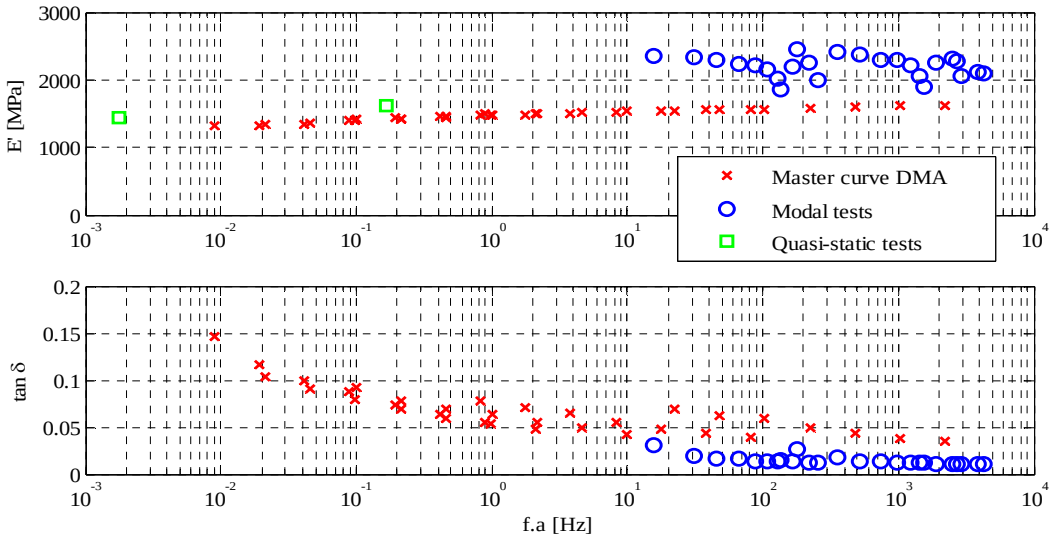

Figure 1. Comparison of experimental methods on E' and tan δ

The order of magnitude of E' at low frequency is respected. The time-temperature equivalence data are consistent with the quasi-static results. At high frequency, there is a gap of about 50 % between the storage modulus prediction and the modal results. This is not observed for the loss factor, for which the comparison is quite conclusive.

CONCLUSIONS

The objective of this study was to highlight the time-temperature equivalence by comparing three experimental methods allowing the identification of the storage modulus over frequency and temperature. This equivalence has been checked on a sample tested with the DMA. Quasi-static tests and modal analysis have been carried out to explore a wider frequency band. Even though the results are rather consistent, the time-temperature equivalence is not yet established. Further investigations are needed to clarify the tBA/PEGDMA behavior. The correlation between quasi-static, DMA and modal analysis remains to be carried out.

REFERENCES

[1] Behl, M. and Lendlein, A., "Shape-memory polymers", Materials Today, 2007, Vol. 10, pp. 20-28
[2] A. Lendlein and S. Kelch, "Shape-memory polymers", Angewandte Chemie International Edition, 2002, Vol. 41, pp. 2034–2057.
[3] Yakacki, et al., "Unconstrained recovery characterization of shape-memory polymer networks for cardiovascular applications, Biomaterials, 2007, Vol. 28, pp. 2255-2263.
[4] Dealy, John and Plazek, Don, "Time-temperature superposition—a user's guide", Rheol. Bull, 2009, Vol. 78, pp. 16-31