HIGH FREQUENCY DYNAMIC MECHANICAL ANALYSIS ON SHAPE MEMORY POLYMERS

P. Butaud[1*] , F. Renaud[2], G. Chevallier[1], M. Ouisse[1] and E. Foltête[1]

[1]FEMTO-ST Applied Mechanics, UMR 6174, CNRS-UFC-ENSMM-UTBM,
24, rue de l'Epitaphe, FR-25000 Besancon
Email: pauline.butaud@femto-st.fr

[2]LISMMA Institut Supérieur de Mécanique de Paris
3, rue Fernand Hainaut, FR-93400 Saint-Ouen
Email: franck.renaud@supmeca.fr

ABSTRACT

This work deals with experimental measurements of the viscoelastic properties of a Shape Memory Polymer (SMP). The material has been previously analyzed using Dynamic Mechanical Analysis in the $[0.1-180]$*Hz frequency range and the* $[0-90]\,^{\circ}C$ *temperature range. In this work, the measurement has been extended to the* $[200-3000]$*Hz frequency range and* $[20-80]\,^{\circ}C$ *temperature range thanks to a High Frequency Viscoanalyzer (HFV). Among the major novelties of this work, this is the first time that this viscoanalyzer is used over the ambient temperature for a full measurement campaign ; the SMP properties have been found over a large frequency and temperature bandwidth without time-temperature superposition (TTS) assumption. Finally a plate with aluminum skins and a SMP core has been designed from the knowledge of the core's behavior, and the model has been experimentally validated. This campaign highlights the uncertainties on the damping properties of the material especially for the lowest loss factor values. It seems that, in these conditions, modal tests combined with identification, might improve the results.*

1 INTRODUCTION

Composite structures are designed to ensure several functions such as stiffness, damping, resistance, mass reduction, thermal or acoustic insulation, etc. To achieve these multiple functionalities, the use of "exotic" materials can be helpful. Shape memory polymers (SMPs) are "smart" materials which have the remarkable ability to recover their primary shape from a temporary one under an external stimulus. SMPs encounter a growing interest over the past ten years, in particular because of their eventual bio-compatibility. They also present many benefits because of their controllable damping property. In this study, the chosen polymer is the tBA/PEGDMA, 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) and the photoinitiator 2,2-dimethoxy-2-phenylacetophenone (DMPA) [1].

In a previous work, the dynamic mechanical characterization of this SMP has been performed using a Dynamic Mechanical Analyzer (DMA50) from Metravib-ACOEM Company on the $[0.1 - 180]$Hz frequency range and $[0 - 90]^\circ$C temperature range. This first experimental campaign highlights promising damping properties controllable by the frequency of the mechanical loadings and the temperature field, see P. Butaud et al. [2]. In order to design a sandwich structure composed of two aluminum skins and a SMP core, the properties have to be extended to larger frequency and temperature domains. This has been first done thanks to the time-temperature superposition (TTS) assumption.

To validate this hypothesis and to improve our knowledge about the SMP properties, a High Frequency Viscoanalyzer (HFV), see F. Renaud et al. [3], has been used to measure the shear properties of the SMP on the $[200 - 3000]$Hz frequency range and $[20 - 80]^\circ$C temperature range. The SMP properties are first identified using a lumped-mass model of the HFV system thanks to Least Mean Square minimization between the test and the simulation results. This allows comparing the results obtained from the DMA and the HFV. Since their operating conditions are different, there are only few couples of temperature-frequency values available, thus the TTS is also used to extend the comparison.

To follow, the SMP datas are used to simulate the behavior of a composite plate constituted with aluminum skins and a SMP core. This sandwich plate has been manufactured and tested in modal analyses. The comparison between tests and simulations highlights distances between the experimental and the simulated modal damping. The updating of the loss factor values is discussed, in section 3.

As a perspective of this work and in order to extend the measurement frequency range of the HFV, one can use a more realistic model that accurately takes into account the eigenmodes. This opportunity is discussed in the paper. To conclude the talk, the design of the HFV and the post-processing improvements will be discussed.

2 SET-UP DESCRIPTION

The High Frequency Viscoanalyzer (HFV), developed by the F. Renaud et al. [3] aims at providing frequency dependent properties of viscoelastic materials over a large frequency bandwidth. This bandwidth starts between $[100 - 200]$Hz and stops between $[2000 - 5000]$Hz according to the stiffness of the specimen.

To measure the viscoelastic properties of the materials, two kinds of setups can be used. Oberst-like setups are based on the analysis of the Frequency Response Function (FRF) of a normalized specimen. This procedure is defined in ASTM-1998 [4] or in ISO-1994 [5]. The latter is generally a multi-layered beam or a multilayered plate constituted with weak viscoelastic layers and stiff elastic layers. The damping properties of the specimen are post-processed from the FRF; its stiffness properties are deduced from the resonance frequency. Unfortunately this method is really efficient if a model, that allows to compute the strain field all over the specimen, is available. Moreover, even if this model is available, it is impossible to ensure an homogeneous strain field in the viscoelastic layer. Thus this method is mainly used to measure the linearized properties. Furthermore, as the signal-to-noise ratio is better close to the resonance frequency, this method only provides confident datas around resonance frequency.

The second kind of setup aims at measuring variations in material properties versus temperature, excitation frequency and imposed strains. It is based on quasi-static excitation, which means that no controllable vibration eigenmodes belongs to the frequency bandwidth of the test. Due to this specificity, it is strongly different to Oberst-like setups. Metravib, MTS or Bose Dynamic Mechanical Analyzer use hydraulic or electromagnetic actuators to load a specimen dedicated to pure traction, compression, shear. The specimen has to be loaded thanks to specific test fixtures dedicated to each kind of test. Our purpose has been to miniaturize such kind of testing device, see Figure 1. The actuators has been replaced by piezo-actuators provided by PhysikInstrumente$^{\text{TM}}$. The test fixture is dedicated to pure shear loading ; to achieve this goal, the setup has three symmetry planes and four specimens, see Dion et al. [6]. Moreover, a bolt is used to preload the setup along the transverse direction. This allows to perform shear measurements according to transverse preload. Six accelerometers are used to measure the accelerations. From these datas, the strain of the specimens are post-processed; two load sensors are used to measure the dynamic shear loading applied to the specimens.

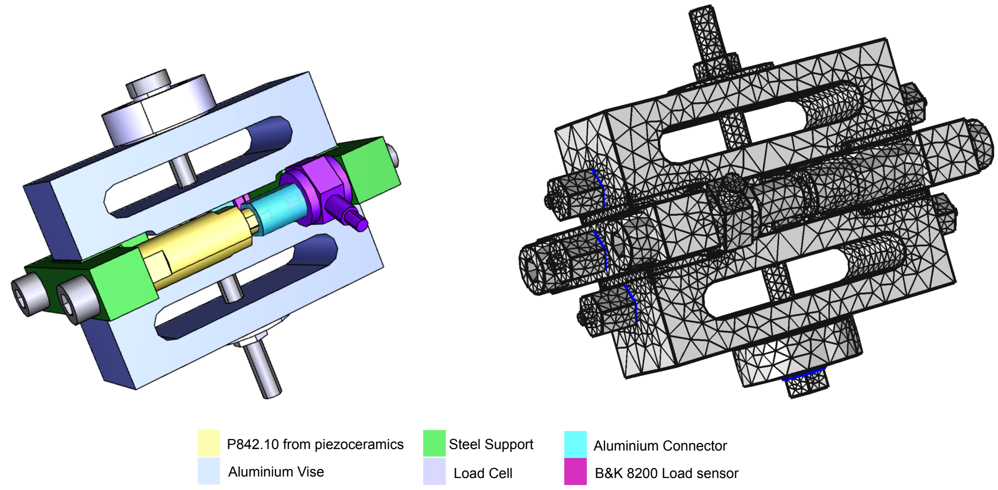

Figure 1: **Left :** 3D CAD view of the HF-DMA. **Right :** Finite Element mesh of the setup : around 10^5 tetrahedron quadratic elements (T10).

2.1 Post-processing of the test

The stress-strain ratio, i.e. the shear modulus, might be computed thanks to the following relationships. If the shear stress σ_{12} is supposed to be uniform in the specimen, it is defined by :

$$\sigma_{12} = \frac{F}{S} \,, \tag{1}$$

where F is the effective load applied to the steel support, see Figures 1 and 2 and S is the section in the plane orthogonal to the shear plane. Assuming that the shear strain ε_{12} and the distortion γ are also uniform in the specimen, one has:

$$2\varepsilon_{12} = \gamma = \frac{u}{t} \,, \tag{2}$$

where u is the effective displacement of each support and t is the thickness of the specimen, see Figure 2.

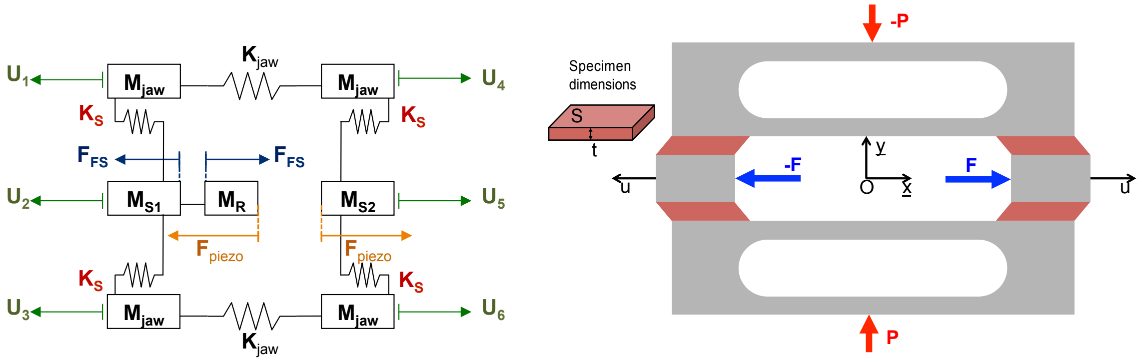

Figure 2: **Left :** Lumped Mass Model of the HFV. **Right :** Dimensions of the specimens. Schema of the test fixture.

The shear modulus is defined as the ratio between the shear stress and the distortion:

$$G = \frac{\sigma_{12}}{\gamma} = \frac{F}{u}\frac{t}{S} \; . \tag{3}$$

However, the shear modulus can not be estimated from the sensors signals processed using Equation (3), because of inertial load effects due to the equipment. The Newton equation applied on the sensor leads to the expression of the stiffness of the specimen:

$$K_S = \frac{\omega^2((M_{S_1} - M_R)\hat{u}_2 + M_{S_2}\hat{u}_5) - 2\hat{F}_{FS}}{2\hat{u}_2 + 2\hat{u}_5 - \hat{u}_1 - \hat{u}_3 - \hat{u}_4 - \hat{u}_6} \; , \tag{4}$$

where $\hat{u}_i$ is the Fourier Transform of the i^{th} accelerometer divided by the excitation angular frequency ω. Using the previous definitions, the shear modulus is defined according to accelerometers and load sensors measurements:

$$G = K_S \frac{t}{S} \; . \tag{5}$$

This post-processing can be done at each frequency of excitation in order to build the frequency dependence of the complex shear modulus $G^*(\omega)$. As shown in Figure 3, the signal is rather good between 200 Hz and 2500 Hz. Below this frequency bandwidth, the acceleration are not large enough and the accelerometers are not sensitive enough, thus the signal is noisy. Above this bandwidth, the first excitable eigenmode (2500 Hz) is visible and prevents direct reading of the phase and modulus.

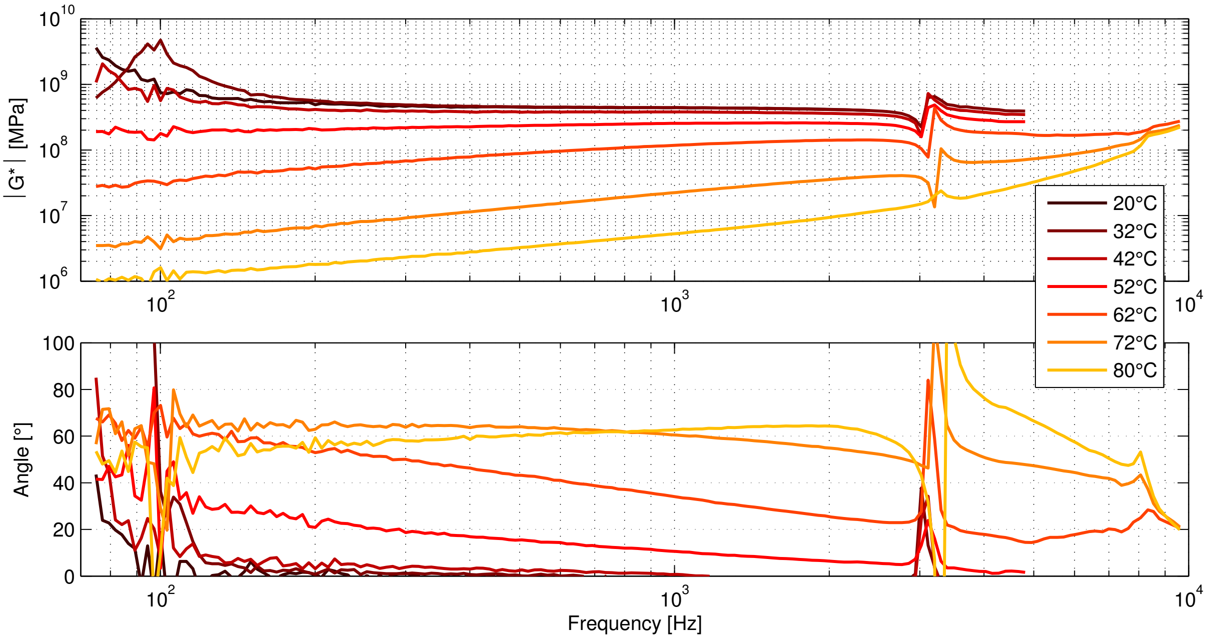

Figure 3: HFV results : absolute value and angle of the complex shear modulus of the tBA/PEGDMA according to the frequency.

A comparison has been done between these HFV measures and those obtained using a Dynamic Mechanical Analyzer (DMA50). The elastic modulus of the HFV results is directly deduced from the shear modulus thanks to the the Poisson's ratio of 0.37 determined in a previous study [7] using a quasi-static test:

$$E^*(\omega) = E\hat{h}^*(\omega) = 2G^*(\omega)(1+\nu) = 2G\hat{h}^*(\omega)(1+\nu) \ , \tag{6}$$

where $\hat{h}^*$ is the constitutive parameter of the viscoelastic model that translate into the frequency dependence of the material. This direct relation could be discussed because of the possible frequency dependence of the Poisson's ratio which is common for polymeric materials [8]. The use of compression measurement and shear measurement in the same frequency bandwidth may answer to this question. Unfortunately, the actual results have been obtained at different frequencies. Figure 4 shows the comparison between the two measurements methods through the master curve obtained by the TTS principle, used to extend the comparable frequency band. These results highlight the fact that both campaigns (DMA-TTS and HFV) provide close results. Looking to the master curves more carefully, we detect the 2500 Hz eigenmode on the right and the noise on the left which make the master curve growing up for each temperature. These artificial values are always in the same frequency ranges and can be easily removed. Moreover, there is a distance between the curves because, for both DMA and HFV tests, it is quite hard to stabilize the temperature. This leads to uncertainties on the storage modulus and on the loss factor, especially when the loss factor variation are really important, around $f.a_T = 10^3$ Hz.

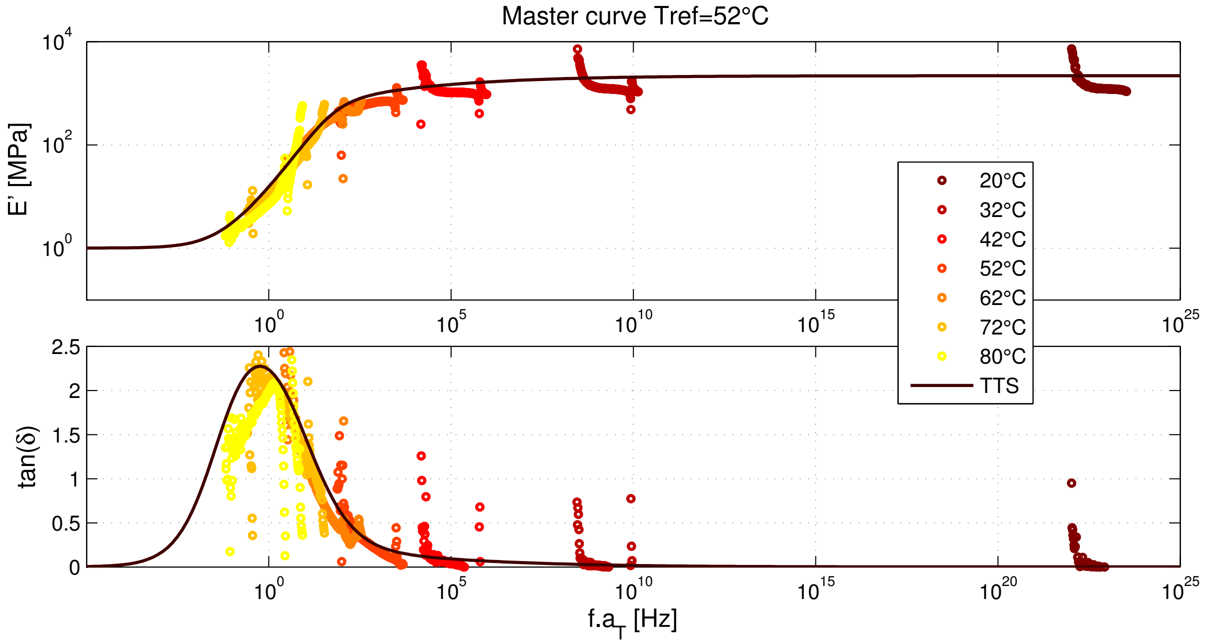

Figure 4: Comparison through the master curve between the HFV results and those obtained by the DMA.

3 MODAL ANALYSIS AS CHARACTERIZATION TEST

In a previous study, it has been shown that a modal analysis is interesting to characterize a SMP in high frequency but it is limited in temperature because of the very low stiffness of the material near the glass transition temperature [9]. An alternative technique is proposed here, by performing modal analysis on a SMP composite structure.
The structure which has been tested is shown in Figure 5. This composite sandwich has been experimentally and numerically studied. The sandwich was in free-free conditions, a broadband random excitation was applied on $[100 - 10000]$ Hz frequency range. The details of the elaboration of the SMP core sandwich and the experimental tests can be found in [10].

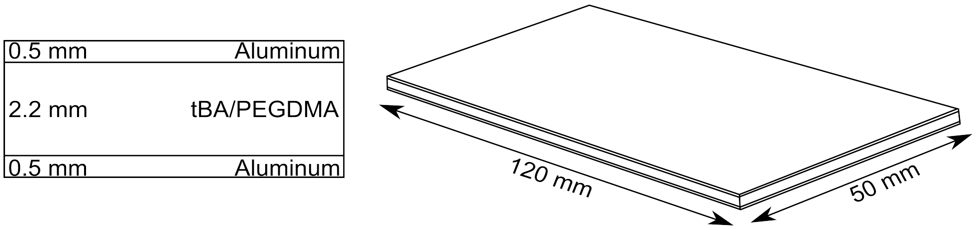

Figure 5. Sandwich structure used in numerical simulation and in experimental tests.

The composite sandwich has been tested between 0 and $130\,^\circ$C every $10\,^\circ$C ($\pm 1\,^\circ$C). Thanks to the aluminum skins which strengthen the structure, the measurements near the glass transition temperature (between $45\,^\circ$C and $75\,^\circ$C according to the frequency) have therefore been possible. Results are obtained experimentally but also through numerical simulations. The simulation parameters are detailed in [10]: the expression of the SMP complex modulus comes from a 2S2P1D model [11] based on the DMA TTS; the mechanical properties of the aluminum skins ($E = 70000$ MPa and $\eta = 10^{-4}$) are taken from the literature [12]. The results of four representative temperatures are presented on Figure 6.

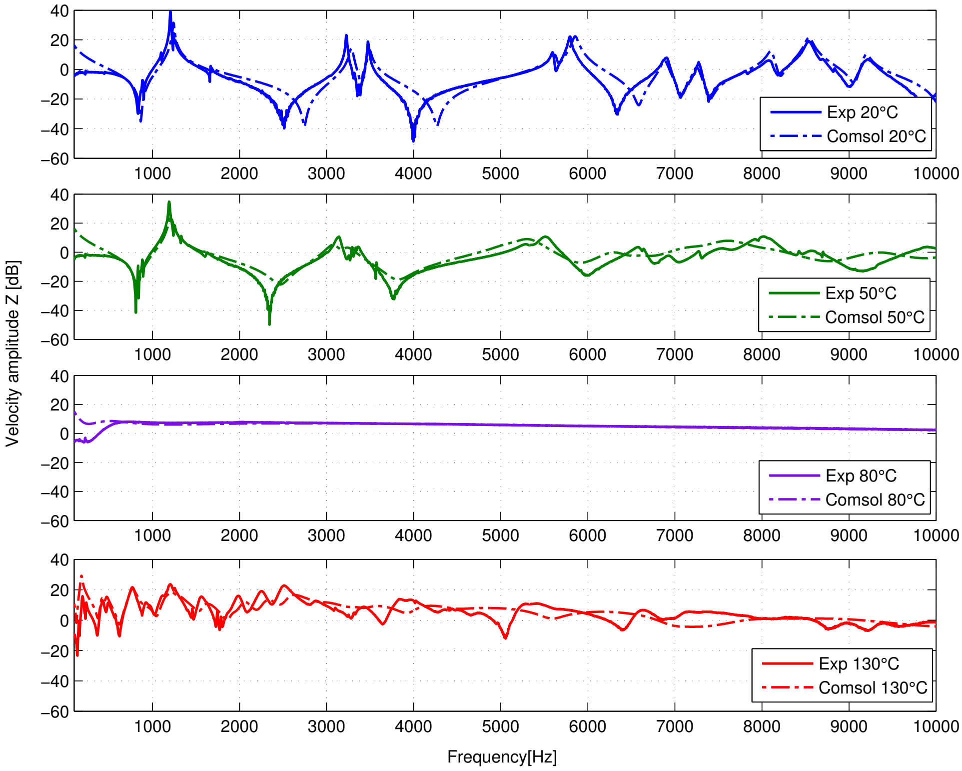

Figure 6. Experimental and simulation results at 20 ° C, 50 ° C, 80 ° C and 130 ° C.

Given the uncertainties on the thickness of the sandwich and on the gluing skins, a good correlation is obtained. A relative distance ε defined by

$$\varepsilon = \frac{1}{n}\sqrt{\sum_{k=1}^{n}\left(\frac{f_k^{simu} - f_k^{exp}}{\frac{f_k^{simu}+f_k^{exp}}{2}}\right)^2}, \tag{7}$$

with n the number of eigenfrequencies measured, is evaluated at 20, 50 and 130 ° C; firstly on the eigenfrequencies, and secondly on the modal damping ratios obtained using a classical modal identification technique. This evaluation was not performed for 80 ° C because of the impressive damping capacities of the SMP which smooths all the resonances. The relative distance on the eigenfrequencies is less than 1% at 20 ° C, and around 3% at 50 and 130 ° C, the Finite Element model with the material datas is so quite representative concerning the stiffness properties of the composite sandwich. The relative distance on the modal damping ratio is less than 10% at 20 ° C but up to 70% for high temperatures. These significant errors on the damping properties can be explained by the large uncertainties on the SMP loss factor values implemented in the FE model. The loss factor values obtained by DMA-TTS are precise at around 50% which is problematic near the glass transition temperature where the loss factor variation are really important; such uncertainties on the loss factor, implemented in the finite element model, permits to bound the modal damping values.

The main conclusions of the comparison between modal tests and simulation based results with DMA datas is that: stiffness in quite well identified from both experimental techniques; damping is harder to identify. To our opinion, the DMA techniques are more suitable in order to identify high loss factors, whereas modal tests are more suitable for lowest loss factors. In order to improve the results, a FEM-based post-processing can be used to identify the material.

4 CONCLUSION

This paper shows results from measurements of viscoelastic properties coming from classical DMA analysis, HFV analysis and Oberst-like analysis. Each device is able to provide temperature dependent results. This is a novelty for the HFV. Let's notice that for the HFV, the temperature range would be extended by the uses of specific piezo actuators. Each device provides interesting results in a specific domain. DMA is able to give results by direct analysis at low frequency. Its results may be extended using TTS. HFV give direct results at higher frequency. Modal analysis can give complementary results for the lowest and highest reduced frequency when the loss factor is small. An ongoing work is to extend the frequency range and reduce the uncertainties by using FEM-based post-processing.

REFERENCES

[1] Christopher Michael Yakacki, Robin Shandas, Craig Lanning, Bryan Rech, Alex Eckstein, and Ken Gall. Unconstrained recovery characterization of shape-memory polymer networks for cardiovascular applications. *Biomaterials*, 28(14):2255 – 2263, 2007.

[2] Pauline Butaud, Morvan Ouisse, Vincent Placet, and Emmanuel Foltête. Experimental investigations on viscoelastic properties of a shape memory polymer. In *ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems*, pages V001T01A029–V001T01A029. American Society of Mechanical Engineers, 2014.

[3] F. Renaud, G. Chevallier, J. L. Dion, and R. Lemaire. Viscoelasticity measurement and identification of viscoelastic parametric models. In *ASME International Design Engineering Technical Conferences and Computers and Information in Engineering Conference*, pages 701–708, Washington DC, 2011.

[4] ASTM E 756-9 : Standard test method for measuring vibration-damping properties of materials, 1998.

[5] ISO 6721-3 : Plastics - determination of dynamic mechanical properties - part 3 - flexural vibration - resonance-curve method, 1994.

[6] JL Dion, G Chevallier, O Penas, and F Renaud. A new multicontact tribometer for deterministic dynamic friction identification. *Wear*, 300(1):126–135, 2013.

[7] Pauline Butaud, Anne Maynadier, Nicolas Brault, Vincent Placet, Morvan Ouisse, Emmanuel Foltete, and Cécile Rogueda-Berriet. Static and dynamic thermo mechanical characterization of a bio-compatible shape memory polymer. In *ICAST 2013, The 24th International Conference on Adaptive Structures Technologies*, pages 1–10, 2013.

[8] Roberto Caracciolo and M Giovagnoni. Frequency dependence of poisson's ratio using the method of reduced variables. *Mechanics of materials*, 24(1):75–85, 1996.

[9] P. Butaud, V. Placet, J. Klesa, M. Ouisse, E. Foltete, and X. Gabrion. Investigations on the frequency and temperature effects on mechanical properties of a shape memory polymer (veriflex). *Mechanics of Materials*, 2015 doi:10.1016/j.mechmat.2015.04.002.

[10] Butaud Pauline, Ouisse Morvan, Foltete Emmanuel, and Rogueda-Berriet Cecile. Smp-based solution for structural damping. In *Conference on Noise and Vibration NOVEM2015*, 2015.

[11] Nur Izzi Md Yusoff, Damien Mounier, Ginoux Marc-Stéphane, Mohd Rosli Hainin, Gordon D Airey, and Hervé Di Benedetto. Modelling the rheological properties of bituminous binders using the 2s2p1d model. *Construction and Building Materials*, 38:395–406, 2013.

[12] F Orban. Damping of materials and members in structures. In *Journal of Physics: Conference Series*, volume 268, page 012022. IOP Publishing, 2011.