# Viscoelastic Properties of PUR Foams

## Impact excitation and dynamic mechanical analysis

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*This work investigates the mechanical properties of polyurethane rigid foams by means of Dynamic Mechanical Analysis (DMA) tests and Impulse Excitation Technique (IET). DMA tests were performed in single cantilever with a sweep in temperature (from -50 °C to 100 °C) and frequency (from 1 Hz to 100 Hz), not determining glass-transition in the test parameter interval. IET tests were used to determine the dynamic modulus of elasticity, showing good accordance with DMA results*

*Keywords: PUR foams, DMA tests, impulse excitation technique*

As a special class of low density materials, cellular solids consist of interconnected networks of cell walls and struts incorporating voids with entrapped gas. Apart from the low specific mass, cellular materials exhibit reduced heat transfer, good impact resistance and good energy absorption [1,2], which recommend them for a wide range of applications, such as thermal insulation, floatability applications, or packaging. Apart from the above mentioned applications, one of the most important uses of rigid foams in particular, from an engineering standpoint, is in structural components of various assemblies as part of sandwich panel cores [1,3-6, 31].

Viscoelasticity is the property of materials to exhibit the characteristics of both solids (through the elastic component) as well as fluids (through the viscous component) [7-9, 31]. Though all materials display viscoelastic behaviour (neither material showing perfect elasticity nor perfect viscosity), the flow component is unnoticeable in most solids while elasticity is unnoticeable in most fluids. In the case of polymers, due to their long chain structure, apart from the evident elastic behaviour, viscous effects determine significant variations in mechanical properties in terms of time (strain-rate sensitivity, creep/stress relaxation) or temperature [7, 10-13].

The viscoelastic response of cellular materials becomes noticeable in such applications where dynamic loadings are predominant [14]. In these scenarios, the classical measures for elasticity (Young's modulus, the shear modulus or the bulk modulus) are insufficient in accurately describing the mechanical behaviour of materials. Instead, the complex modulus $M^*$ should be used, which is described as [7]:

$$M^* = M' + i \cdot M'' \quad (1)$$

where $M'$ is the storage modulus (which represents a measure of the stored elastic potential), $M''$ is the loss modulus (a measure of the energy dissipated through viscous effects) and $i$ is the imaginary unit [7,15]. A couple of methods of determining the above mentioned material characteristics are through Dynamic mechanical analysis (DMA) and Impulse excitation technique (IET).

DMA tests have long been used in the viscoelastic characterization of polymers and consist of applying a sine deformation to a specimen in various loading patterns (most common being single cantilever, double cantilever, shear [16], tension [17,18] or three-point bending [19]) at varying frequencies and temperatures [20,21]. Due to the viscous nature of the polymers, a difference in phase between the applied strain and the measured force will be noticed with the help of which the components of the complex modulus will be determined [15].

In literature, DMA studies were performed on various cellular materials with different parameters, most emphasis being placed on temperature dependency. Rodríguez-Pérez et al. investigated the variation of the dynamic moduli of polyolefin foams with temperature by performing a temperature sweep between 125 and 240°C [20]; Da Silva et al. investigated modified polyurethane foams in torsion with a temperature sweep between -50 and 130°C; Chattopadhyay et al. analyzed the viscoelastic behaviour of the polyurethane-imide/clay hybrid coatings in nitrogen atmosphere in shear mode in a temperature range from -30 to 200°C [16]. In terms of frequency influence, Kanny et al. tested PVC foams in three-point bending with densities ranging from 75 to 300 kg/m$^3$, the dynamic moduli showing little variation over the tested frequency range (1-10 Hz) [19] while Saint-Michael et al. studied the effect of filler size in DMA tests on rigid polyurethane foams reinforced by mineral fillers at low frequencies (from $10^{-5}$ up to 5 Hz) and a temperature range of -170 up to 400°C [22].

Another way of determining the dynamic moduli of materials is with the help of Impulse Excitation Technique. Though not as comprehensive as DMA tests, the advantages of this method consist of the simple test setup, reduced costs and relative short analyses while it enables the determination of the Poisson ratio and of possible anisotropy.

The aim of this study is to determine the variation of dynamic properties with temperature, frequency and density for a rigid polyurethane (PUR) foam, the range of

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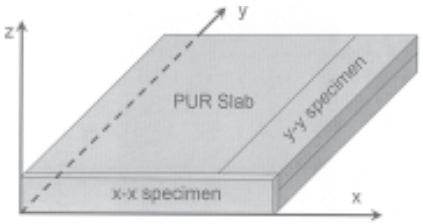



Fig. 1. Specimen manufacturing for anisotropy study

variation of test parameters being chosen according to the usability envelope of the material for the above mentioned applications (a temperature range of -50 °C to 100 °C and a frequency variation between 1Hz and 100 Hz). Three densities were tested: 100 kg/m$^3$, 145 kg/m$^3$ and 300 kg/m$^3$. DMA results were compared with IET results in order to validate the latter method. The gathered experimental data was used for designing a viscoelastic material for finite element analysis simulations.

## Experimental part

*DMA Tests*

DMA tests were performed on a machine Q800 by applying a sinusoidal strain in dual cantilever on 60 mm x 10 mm x 4 mm prismatic specimens cut along two directions, as presented in Figure 1.Atemperature sweep was performed from -50 °C to 100 °C with a 10 °C step and for 7 frequencies: 1, 3, 6, 10, 30, 60 and 100 Hz, each frequency being tested on a given temperature step.

Figure 1 presents the results for density of 100 kg/m$^3$, figure 2 the results for145 kg/m$^3$ and figure 3 the results for 300 kg/m$^3$, in each case showing the variation of the storage modulus (a), loss modulus (b) and damping coefficient (c) with frequency for the specimens cut along the Y-Y axis.

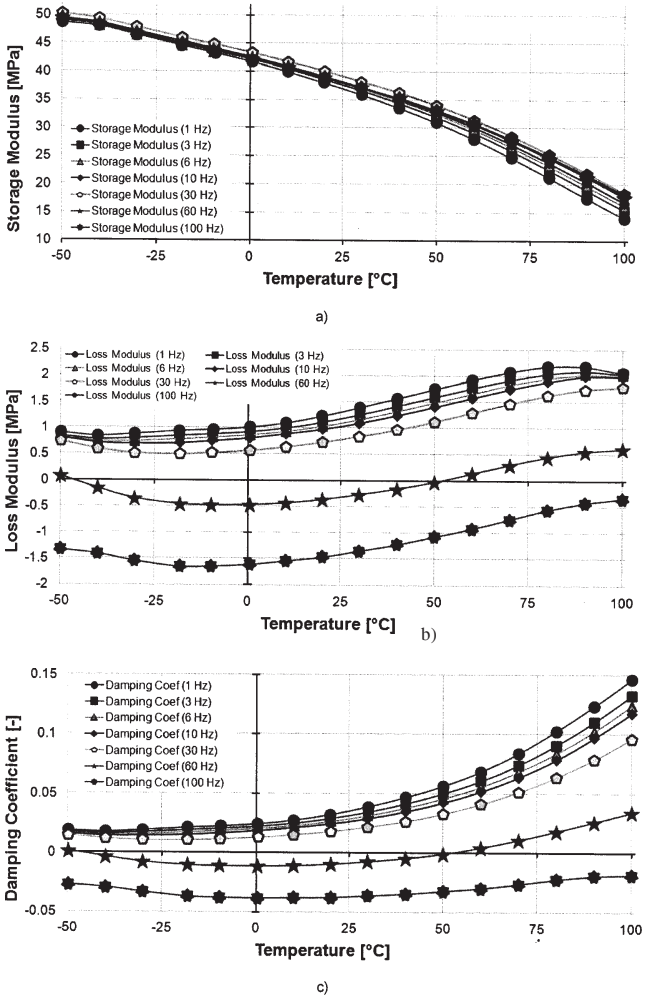



Fig. 2. Variation of (a) storage modulus, (b) loss modulus and (c) damping coefficient with frequency for the 100 kg/m$^3$ density

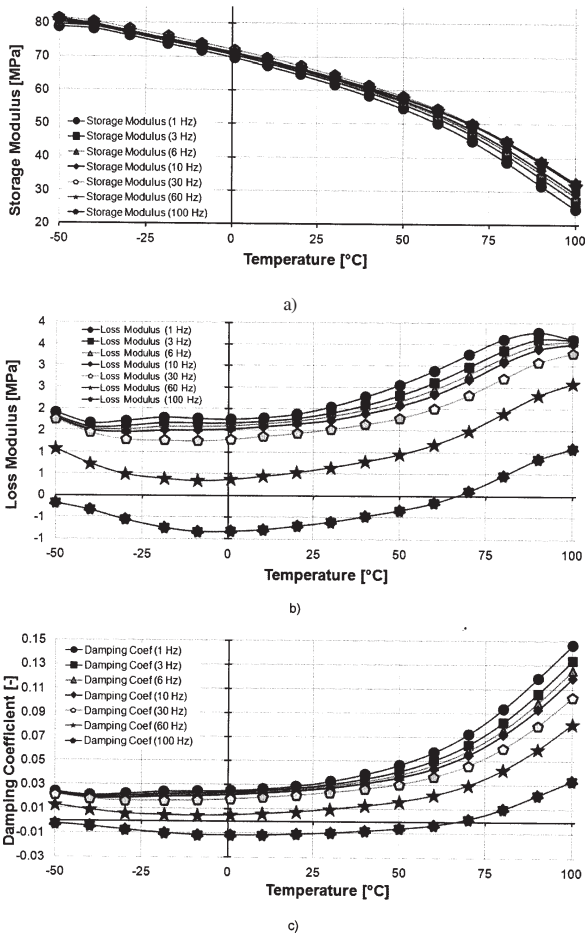



Fig. 3. Variation of (a) storage modulus, (b) loss modulus and (c) damping coefficient with frequency for the 145 kg/m$^3$ density

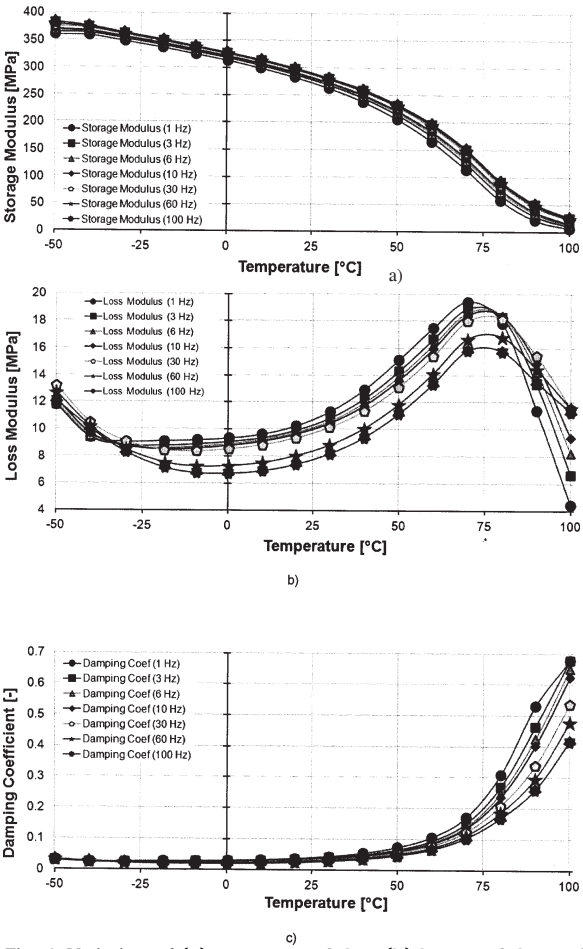



Fig. 4. Variation of (a) storage modulus, (b) loss modulus and (c) damping coefficient with frequency for the 300 kg/m$^3$ density

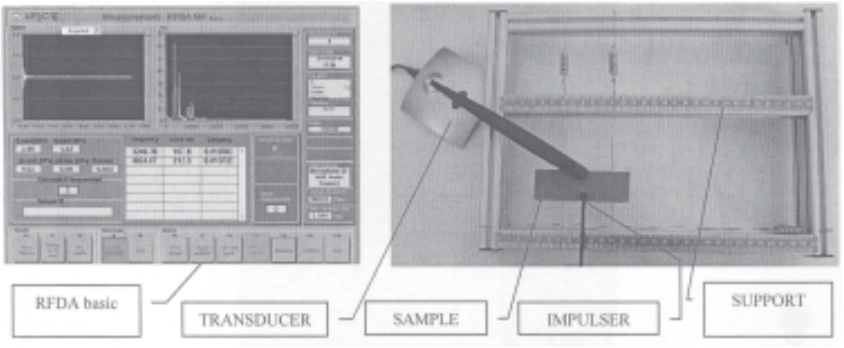


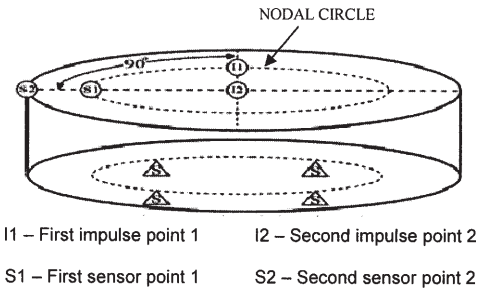

Fig. 5. Experimental setup for the impulse excitation method

*Impulse excitation tests*

The impulse excitation test method measures the fundamental resonant frequency of test specimens by mechanically exciting them with a singular elastic strike with an impulse tool. The induced vibration's energy is dissipated in the material and it has a frequency spectrum according to its resonant frequencies which are dependent on several material parameters such as the elastic response, the geometry and the density [23,24]. Each frequency will damp according to the energy absorption of the material. The mechanisms for damping through internal friction or mechanical loss is characteristic for each microstructure and thus varies from one class of materials to the other.

In order to determine elastic properties with this technique, a non-destructive testing device named Resonant Frequency and Damping Analyser (RFDA) was used. It consists of an aluminium grid that supports a number of polyamide strings on which the specimen is placed. A mechanical impulse is induced in the specimen with the help of a steel sphere of 6mm diameter encastred at the end of a 100 mm flexible polymeric rod. The induced vibrations are detected using a non-contact transducer (microphone) and the acquired data is processed by specialized computer software [25]. The experimental setup is presented in figure 5.

The elastic modulus was determined on round samples by registering the first and second natural vibrations, the setup of the support, impulse and sensor points' being presented in figure 6.




I1 – First impulse point 1 I2 – Second impulse point 2

S1 – First sensor point 1 S2 – Second sensor point 2

Fig. 6. Support, impulse and sensor points for first and second natural vibration

For the first natural vibration mode, the nodes are located along two orthogonal diameters; offset 45° from the point where the vibration was induced. The anti-nodes are located along two orthogonal, 90° offset, diameters in the disc, with one diameter intersecting the point where the vibration was induced. For the second natural vibration mode, the nodes are located in a circle concentric with the centre of the disc with fractional radius of 0.681 of the disc radius. The anti-nodes are located at the centre and around the circumference of the disc specimen.

The Poisson's Ratio can be determined directly from the experimental values of the first natural resonant frequency ($f_1$) and the second natural resonant frequency ($f_2$), the value for Poisson's Ratio is interpolated using the ratio of the second natural resonant frequency to the first natural resonant frequency ($f_2/f_1$) is correlated with the ratio of the specimen thickness to the specimen radius ($t/l$).

For the elastic Modulus of round disc samples, two values of Young's modulus are calculated ($E_1$ and $E_2$)from the two resonant frequency measurements (eq. 2 and 3), the final value of Young's Modulus being determined as their average (eq. 3).

$$E_1 = \frac{\left[37.6991 \cdot f_1^2 D^2 m\left(1-\mu^2\right)\right]}{K_1^2 t^3} \quad (2)$$

$$E_1 = \frac{\left[37.6991 \cdot f_2^2 D^2 m\left(1-\mu^2\right)\right]}{K_2^2 t^3} \quad (3)$$

$$E = \frac{E_1 + E_2}{2} \quad (4)$$

where $E$ is Young's Modulus (MPa), $E_1$ is the first natural evaluation of Young's modulus, $E_2$ is the second natural evaluation of Young's modulus, $f_1$ and $f_2$ are the first and second natural resonant frequencies of the disc (Hz), $D$ is the diameter of the disc (mm), $m$ is the mass of the disc (kg), $\mu$ is the Poisson's Ratio, $K_1$ and $K_2$ are the first and second natural geometric factors, $t$ is the thickness of the disc (mm) and $r$ is the radius of the disc (mm)

In order to compare the elastic modulus determined by impulse excitation technique to the values obtained by means of DMA tests, the complex modulus must be determined from DMA data, with the help of equation (5)

$$M^* = M' + i \cdot M'' = \sqrt{M'^2 + M''^2} \quad (5)$$

The comparison between the impulse excitation results and the DMA results for the three densities is presented in table 1, IET determining higher values, between 6.7 and 27.7%.

**Results and discussions**

The results of the anisotropy study show that, despite the similarity in variation pattern of the recorded properties, the specimens cut along the X-X direction exhibit lower properties than the ones cut along the Y-Y direction, showing average variations of around 16% for the density of 100 kg/m$^3$, 5% for the density of 145 kg/m$^3$ and 8 for the density of 300 kg/m$^3$. Such a difference in the variation of storage and loss modulus with temperature at 10 Hz is presented in figure 7 for the 100 kg/m$^3$.

Considering the aspect presented in experimental part as well as for other tests performed on this material [26-29] it can be observed that the PUR foam with 300 kg/m$^3$ density has a different behaviour compared with the other two densities. This behaviour can be associated with the different types of structure of the PUR foam at different densities. Figure 4 presents SEM imagery of the three

| Density [kg/m$^3$] | Complex Modulus [MPa] | | | | | Variation [%] | |
|---|---|---|---|---|---|---|---|
| | Impulse excitation technique | DMA | | | | | |
| | | Y-Y direction | | X-X direction | | Y-Y direction | X-X direction |
| | | 1 Hz | 100 Hz | 1 Hz | 100 Hz | | |
| 100 | 45.46 | 41.79 | 43.41 | 35.16 | 36.01 | 6.71 | 27.75 |
| 145 | 72.44 | 65.82 | 67.76 | 62.43 | 64.51 | 8.45 | 14.13 |
| 300 | 324.94 | 282.96 | 301.21 | 260.41 | 278.89 | 11.24 | 20.50 |

**Table 1**
RESULTS FOR THE YOUNG'S MODULUS FOR THE THREE DENSITIES

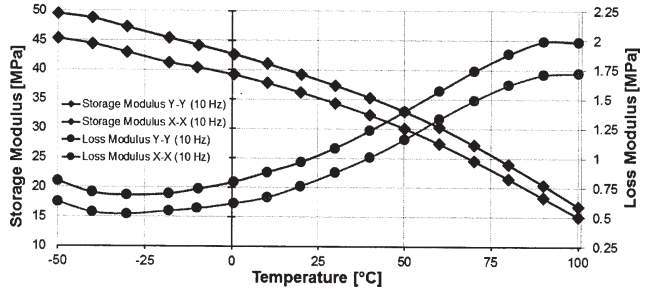



Fig. 7. Storage modulus and loss modulus variation with temperature (at 10 Hz) for the specimens cut along the X-X and Y-Y directions

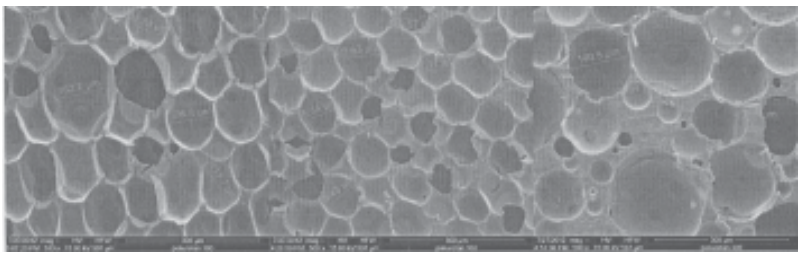


Fig. 8. SEM images of PUR foams with measured cell diameters for a) 100 kg/m$^3$ density, b) 145 kg/m$^3$ density and c) 300 kg/m$^3$ density

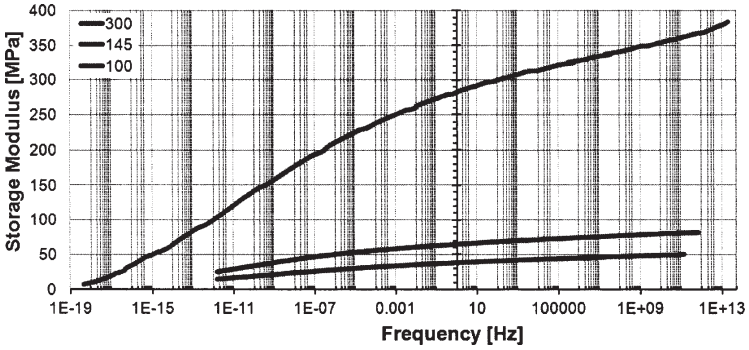



Fig. 9. Time-temperature superposition for the threedensities

investigated densities which shows that the structure of the 100 kg/m$^3$ and 145 kg/m$^3$ densities can be considered that of a cellular material while the structure of the 300 kg/m$^3$ resembling more a porous solid.

The experimental programme consisting of DMA tests with a temperature sweep from -50°C to 100 °C was not able to determine the glass transition temperature of the studied PUR foams. Literature studies suggested a glass transition temperature of approximately 150 °C [30], this temperature being, however, outside the usability envelope of the investigated materials.

Regardless of the foam density, frequency has a smaller effect on the viscoelastic parameters than temperature: the largest variation in storage modulus with frequency was observed for the 145 kg/m$^3$ density at 100°C: 50% increase from 1 Hz to 100 Hz, from 25 MPa to 39 MPa; the largest variation in storage modulus with temperature was observed for the 300 kg/m$^3$ density: 1300% increase for a drop in temperature from 100 to -50°C, from 25 MPa to 384 MPa.

For several cases of low temperature (-50 ..-70 °C) and high frequencies (60 and 100 Hz), the damping coefficient becomes negative, which corresponds to an unstable propagation of oscillations in the material, thus gaining driving properties instead of damping (self-excitation) [31].

The time-temperature superposition for the three densities is presented in figure 9.

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