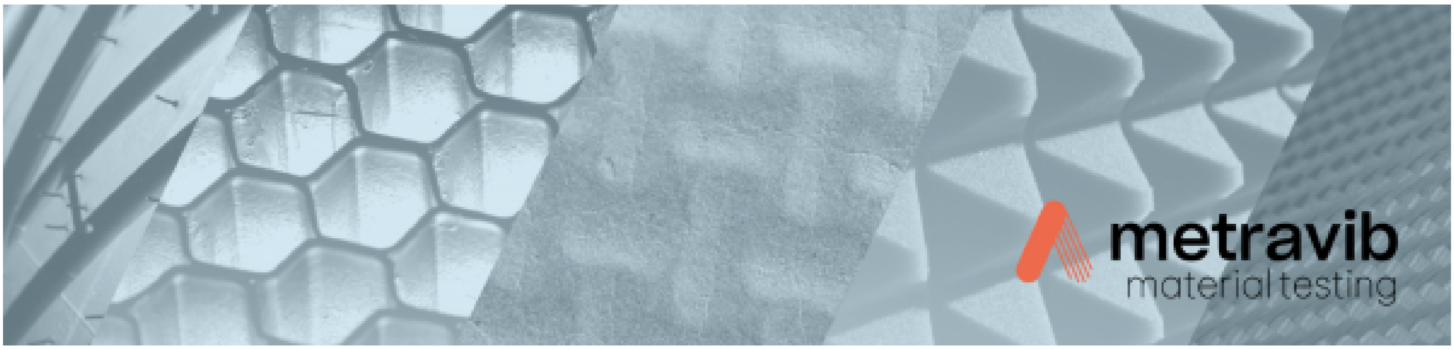


Control and Measurement Periods in Dynamic Mechanical Analysis

Keywords: DMA, Viscoelastic, Materials, control, Polymers

Figure 1. Metravib DMA+300

Introduction

Dynamic Mechanical Analysis (DMA) is largely used to characterize the viscoelastic properties of polymeric materials as they involve both solid and liquid-type characteristics. In DMA, a sinusoidal load (stress or strain) is applied to a sample and the corresponding response (strain or stress) is recorded [1].

When a sinusoidal stress is applied, it is described by the equation:

$$\sigma = \sigma_0 sin\, \omega t$$

where σ is the stress at time t, σ_0 is the amplitude of the stress and ω is the angular frequency.

The resulting strain is as well sinusoidally shaped:

$$\gamma = \gamma_0 sin\,(\omega t + \delta)$$

where γ is the strain at time t, γ_0 is the amplitude of the strain and δ is the phase difference between the stress signal and the strain signal.

For ideal elastic solids respecting the Hooke's law, this phase lag δ is 0, whereas, for perfect viscous solids respecting Newton's law, this phase lag is 90°. Accordingly, a complex modulus is defined as the combination of elastic part (E') and the viscous part (E"):

$$E^* = E' + iE''$$

$$E' = E^* \cos\delta \text{ and } E'' = E^* \sin\delta$$

where E' is the elastic (storage) modulus, representing the stored energy by the sample, and E'' is the viscous (loss) modulus, representing the dissipated energy by the sample. The loss factor (damping) is defined as the ratio between the loss modulus and the storage modulus:

$$\tan\delta = \frac{E''}{E'}$$

In terms of energy, the loss factor represents the ratio between the dissipated and the stored energy.

While understanding the basics of how DMA functions, it is also important to understand how Metravib's DMA equipment regulates the imposed sinusoidal load (stress or strain). This understanding of the stabilization process could help operators to avoid noisy results.

The steps DMA takes from generating the imposed signals to the measurements are as follows:

1. It generates the signal of relative amplitude without overshooting.
2. Stabilization of the generated signal.
3. Regulates and sees if the generated and stabilized signals are of the same amplitude as the operator's input.
4. If yes, it takes the measurement. If not, it regenerates the signal of amplitude closer to the operator's input.

Figure 2 shows the representation of the above 4 steps in pictorial form.

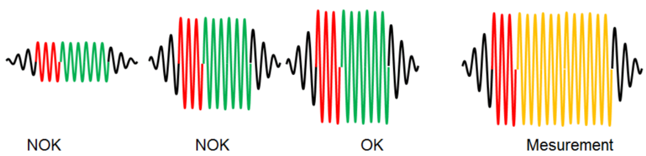


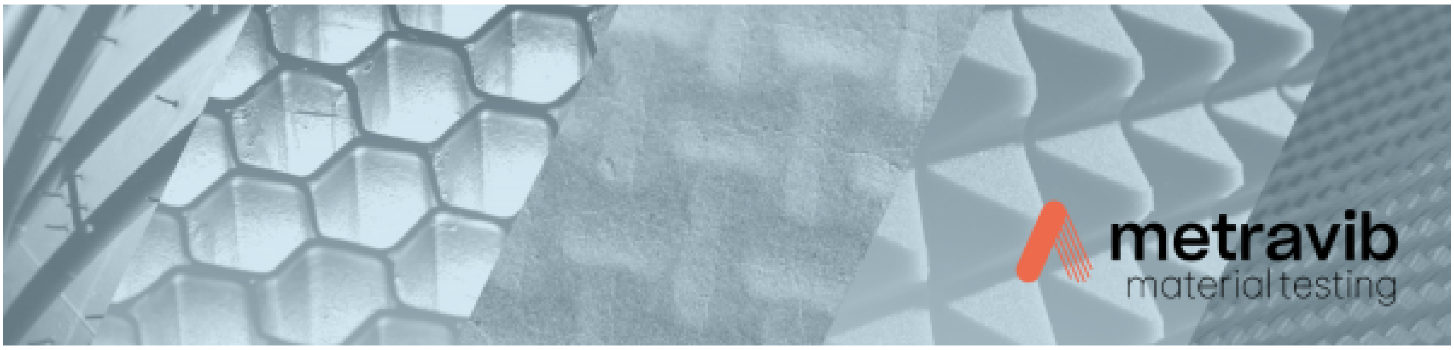
Figure 2. Pictorial representation of 4 steps from generating signals to the measurement. Generate (Black), Stabilize (Red), Regulate (Green) and Measurement (Yellow).

The aim of this study is to show the impact of different control parameters (which can be easily set-up in the Metravib software) on the results quality.

Materials & methods

The material tested for this study is polystyrene foams. This type of foam is commonly used across many industries, such as the shoe industry. The specimens used were of cuboidal shape with dimensions of 10 x 10 x 20 mm.

The specimens were studied in compression mode Figure (3). The specimens were glued to



make sure that the specimen stayed in contact with the specimen holders. The dynamic displacement of 10 µm was applied on the specimen. This test was performed at 1 and 3 Hz.

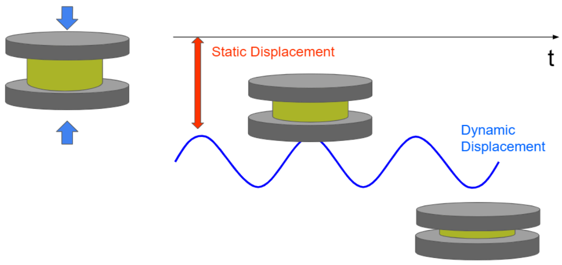


Figure 3. Schematic diagram of Compression mode

Dynamic displacement (µm)	10
Frequency (Hz)	1 and 3
Temperature (˚C)	-140 to 100
Rate (˚C/min)	2
DMA	DMA+300
Test Mode	Compression
Mesure 1 (Fig 4)	control step - 6 periods Measurement step - 12 periods
Mesure 2 (Fig 5)	control step - 30 periods Measurement step - 60 periods

Table 1. Test parameters

The test was performed twice using the same parameters mentioned in Table 1. Whereas, the number of periods/cycles to regulate the applied displacement and measure the properties of the foam were changed during the second test.

Results and Discussion

Figure 4 shows the results obtained for the temperature ramp at 1 and 3 Hz, for the standard control periods. The results are presented in terms of storage modulus (E') and loss factor (tan δ) against temperature. The decrease of E' as a function of temperature indicates the molecular dynamic rearrangements of elastomer chains between the glassy state (localized molecular movement) and the rubber-like state (high amplitude movement) [2].

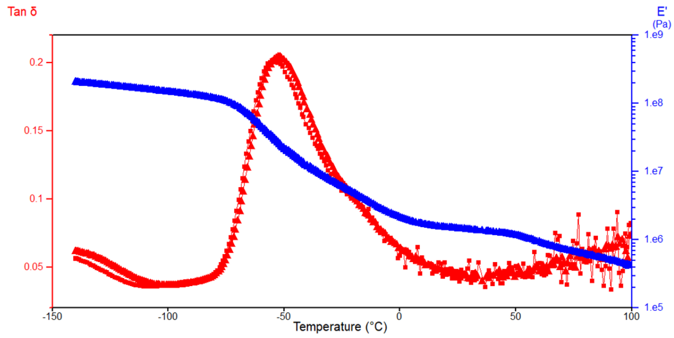
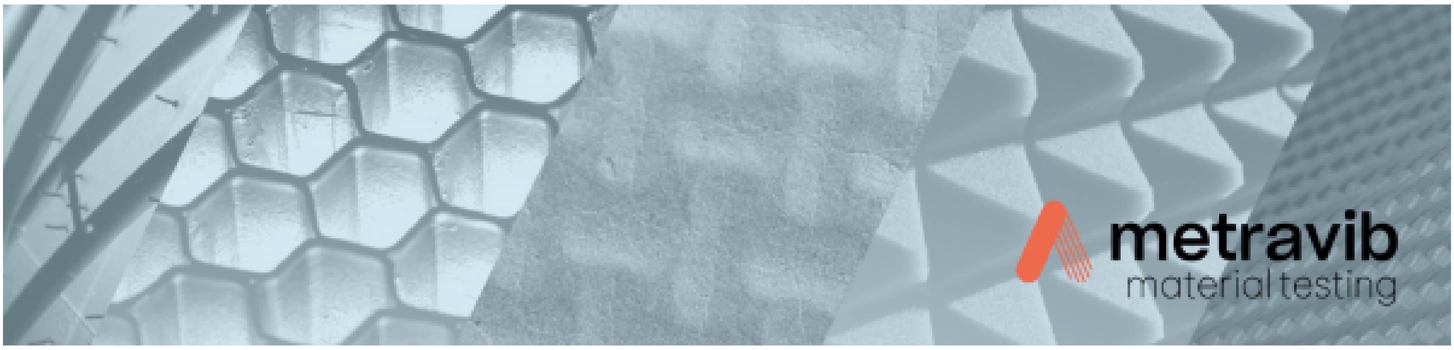


Figure 4. E' and tan δ as a function of temperature at 1 and 3 Hz with standard control and measurement periods

Figure 5 shows the results obtained for the temperature ramp at 1 and 3 Hz. For this test, the control periods were changed to 30 instead of 6, and measurement periods were changed to 60 from 12, significantly increasing the noise/signal ratio.



The results obtained from the second test were less noisy at high temperatures.

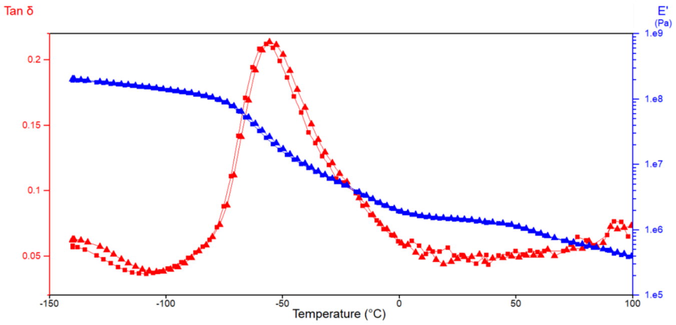


Figure 5. E' and tan δ as a function of temperature at 1 and 3 Hz after changing the control and measurement periods

Conclusions

In this study, two temperature ramp tests were performed on the same polystyrene foam at 1 and 3 Hz. The control and measurement periods were kept different for the two tests to see the effect of these periods on the viscoelastic results in terms of noise.

From figure 4 and 5, it was observed that increasing the number of control and measurement periods have significantly decreased the noise on the results.

Optimizing the quality of a DMA measurement requires a detailed understanding of how the characterization instrument works. The Metravib software enables the user to set advanced parameters to guarantee accurate and reproducible results.

References

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