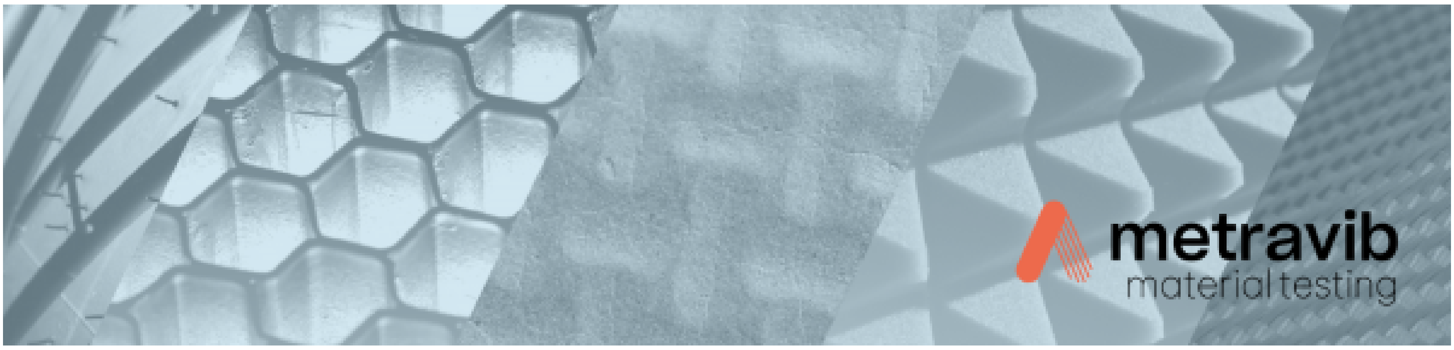


Effect of Temperature Increment on DMA Measurements

Keywords: DMA, Glass Transition, Temperature rate, Viscoelastic properties, Temperature sweep

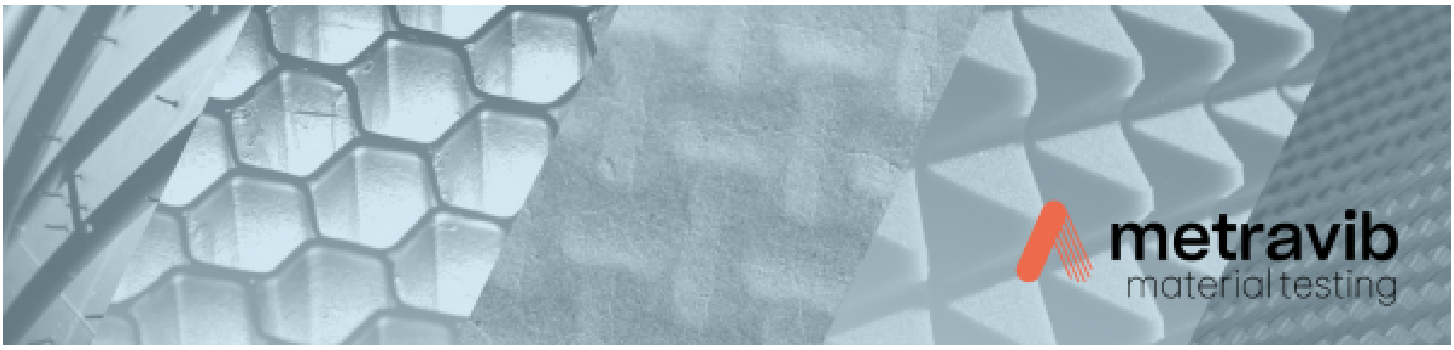
Figure 1. Metravib DMA+1000.

Introduction

Dynamic Mechanical Analysis (DMA) measurements are used to study and characterize the viscoelastic behavior of polymer like materials. To characterize the material, a sinusoidal stress is applied and the strain induced is measured. One of the widely used methods in industries, is to study the materials behavior as a function of temperature. This method is called temperature sweep.

Temperature sweep is a known method that involves measuring the viscoelastic behavior at constant frequency while varying the temperature of the sample. During the temperature sweep measurements, it is important to know that the different temperature rates from one temperature to another shows a significant difference in viscoelastic properties. This could also induce a difference in the glass transition peak of the material.

This study was performed to particularly show the effect of temperature rate during a



temperature sweep on the viscoelastic behavior of the materials. Two different morphologies were used for better understanding.

Materials & methods

The material tested is an EPDM (Ethylene Propylene Diene Monomer). EPDM is a type of durable synthetic rubber, commonly used across many industries, such as Tire Industries. The specimens used were (1) the standard Goodrich Cylindrical Blocks, i.e., diameter - 17.9 mm and height - 25 mm and (2) a strip of 2 mm thickness and 15 mm width.

The specimens (Goodrich) were studied in compression mode (see Figure. 2) whereas the strip is in tension mode (see Figure. 3).

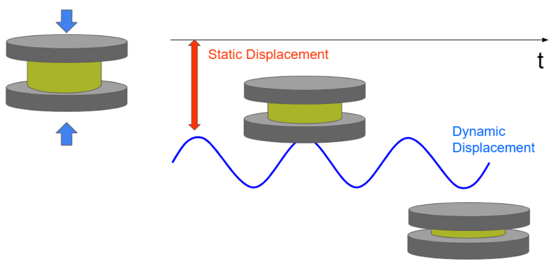


Figure 2. Schematic diagram of Compression mode

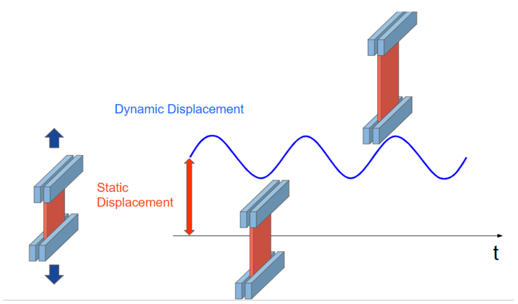


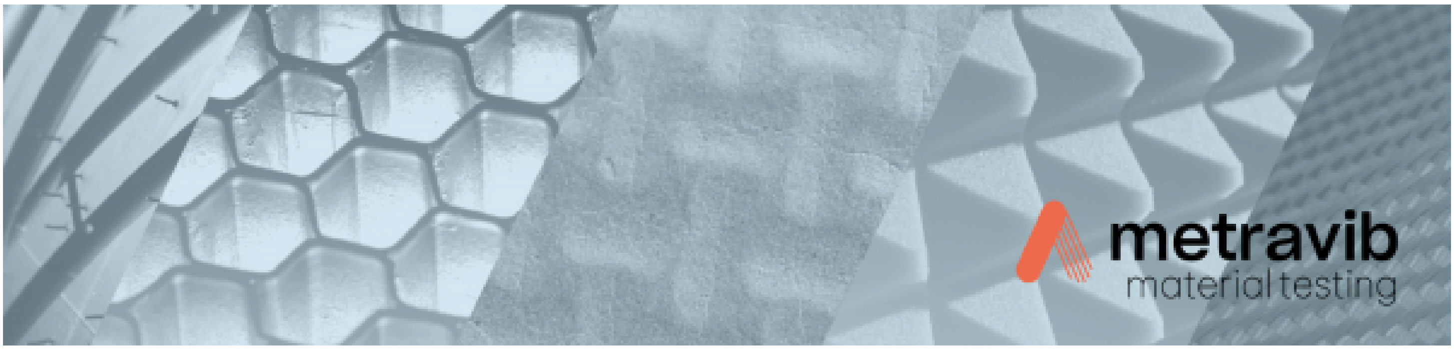
Figure 3. Schematic diagram of Tension mode

Since, the goal is to demonstrate the effect of temperature rate on a material's viscoelastic behavior, a temperature sweep (-70˚C to 13˚C) was performed at 10 Hz. Three measurements were performed with three different temperature rates for each mode. The parameters used for the measurements are mentioned in Table 1.

Parameters	**Compression**	**Tension**
Dynamic Force /displacement	1 N	2e-5 m
Frequency (Hz)	10	10
Static Force/displacement	-5 N	4e-5 m
Temperature (˚C)	-70 to 13	-70 to 13
Rate (˚C/min)	10, 5 and 2	10, 5 and 2
DMA	DMA+1000	DMA+1000

Table 1. Test parameters

The cold source used to go to -70˚C was Metravib's Air Chiller System. This air chiller system can help perform measurements until -70˚C and save some money on liquid nitrogen.



Results

Compression:

Figure 4 shows the results obtained for the temperature sweep at three different temperature rates i.e., 10, 5 and 2˚C/min, in compression mode. The results are presented in terms of storage modulus (E') and loss factor (tan δ) as a function of temperature.

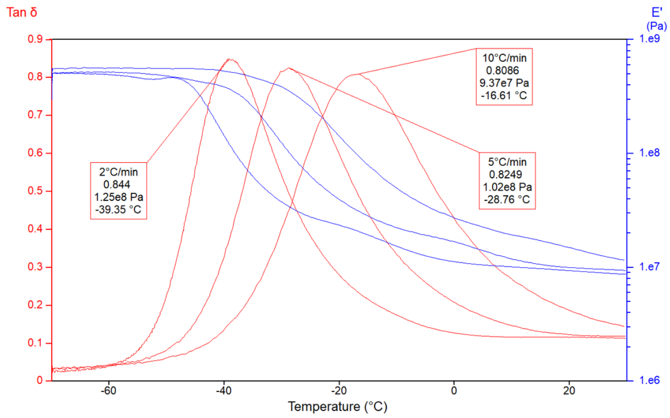


Figure 4. E' and tan δ as a function of temperature at 10 Hz at three different temperature rates i.e., 2, 5 and 10 ˚C/min, for goodrich specimen.

In Figure 4, it can be observed that the change in temperature rate has an effect on the glass transition temperature of the material i.e., an increase in glass transition temperature with increasing temperature rate was observed. The glass transition temperature increased from ~-39˚C at 2˚C/min to ~-17˚C at 10˚C/min.

Tension:

Figure 5 shows the results obtained for the temperature sweep at three different temperature rates i.e., 10, 5 and 2˚C/min, in compression mode. The results are presented in terms of storage modulus (E') and loss factor (tan δ) as a function of temperature.

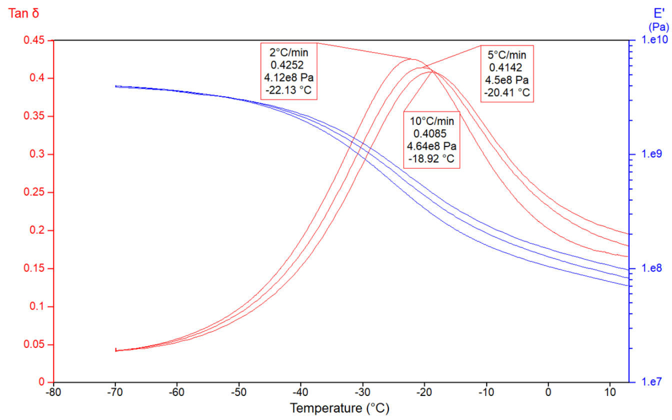
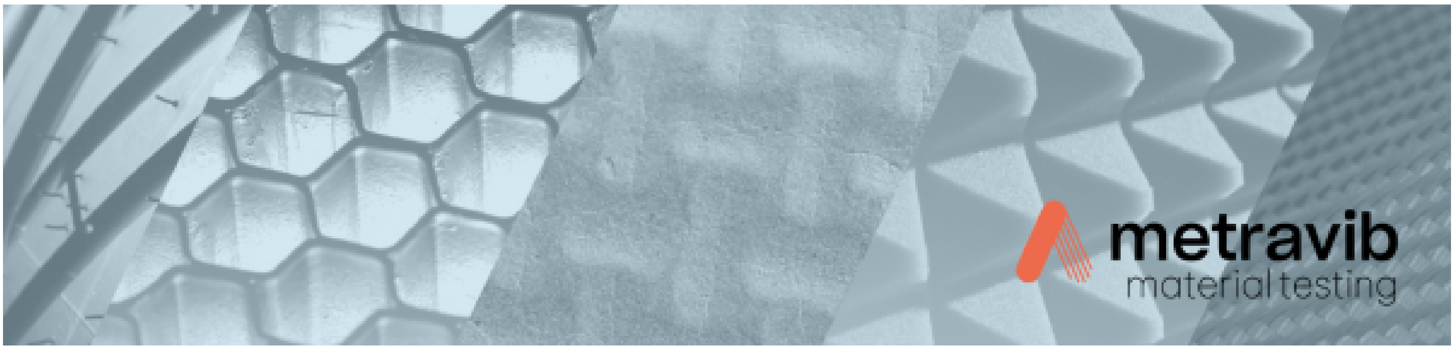


Figure 5. E' and tan δ as a function of temperature at 10 Hz at three different temperature rates i.e., 2, 5 and 10 ˚C/min, for strip specimen.

In Figure 5, increase in glass transition temperature with increasing temperature rate was also observed. The glass transition temperature increased from ~-22˚C at 2˚C/min to ~-19˚C at 10˚C/min.

The change in glass transition temperature was found to be relatively significant in goodrich specimen than in strip specimen, as a result of the volume difference. This change in glass transition in both modes could be associated



with the fact that the temperature distribution in the sample is not even close to homogenous, which makes the polymeric chains behave differently.

Conclusions

This study shows the precision of Metravib's DMA to characterize elastomer materials with different temperature rates. The temperature sweep performed at different temperature rates has brought important information on the effect of temperature rates on glass transition temperature of specimen of different volumes. Moreover, the behavior of storage modulus (E') and loss factor (Tan δ) was studied as a function of temperature. The study shows that the glass transition temperature increases with the increasing temperature rate.

An important point to clarify in DMA: kinetic measurements, such as a temperature sweep, necessarily generate measurement uncertainty (the modulus of E', E" or tan delta in absolute values can only be obtained under stabilized conditions on temperature stages). The faster the temperature increases or decreases, the more important it is.

This fundamental aspect of measurements with temperature sweeps should also be considered in relation to the thermal stabilization steps to be implemented before starting a ramp.

For instance, for a Goodrich specimen, Metravib recommends a 30 minutes stabilization time at low temperature (-70˚C in our case) before starting the temperature ramp at 2˚C/min.

Out of all the temperature rates, the suggested temperature rate by Metravib for temperature sweeps is 2˚C/min as per the ASTM and ISO standards.

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