# Thermo-mechanical characterisation of viscoelastic materials by dynamic measurements of the complex shear and bulk moduli

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## 1 Introduction

The behaviour of homogeneous isotropic materials is described by two elastic constants (taken generally among the Young modulus $E$, the shear modulus $G$, the bulk modulus $K$ and the Poisson's ratio $\nu$), which are frequency- and temperature-dependent in the case of viscoelastic materials. In a theorical study, Guillot and Trivett [1] show that measurements of the pair $(G,K)$ or $(E,K)$ are best suited for the determination of the remaining elastic constants, such as the Poisson's ratio. However, in practice, the bulk modulus and the Poisson's ratio are often considered as independent of temperature and frequency. We propose in this work to study the validity of this hypothesis by measuring independently the shear and the bulk moduli of an amorphous synthetic rubber (Deltane 350 from Paulstra®)as a function of temperature and frequency.

## 2 Measurements

Dynamical Mechanical Analysis in a double lab shear configuration is used to measure the complex shear modulus of Deltane 350. Specimen of size 9mm $\times$6mm $\times$2mm are tested in the frequency range $0-400$Hz, and over the temperature range $-40^o$C to $43^o$C with a heating rate of $2^o$C/min. A dynamic displacement of 5$\mu$m is applied to the specimen to remain in the linear viscoelastic domain.
The measurement of the complex bulk modulus is carried out on a Schenck VHF7 servo-hydraulic machine. Cylindrical specimen of diameter 14mm are tested under a hydrostatic pressure of 20 bar, in the frequency range $3-200$Hz, and from $-30^o$C to $23^o$C. A dynamic displacement of 10$\mu$m is considered to remain in the linear viscoelastic domain.

## 3 Analysis

The time-temperature superposition principle is applied to measurements of shear and bulk moduli in order to obtain the master curves of the synthetic rubber at $12^o$C. In this work, both horizontal and vertical shift coefficients are applied to the measured isotherms of storage and loss (shear and bulk) moduli. Those shift coefficients are calculated using a method based on the Kramers-Kronig relations, described in detail in [2]. The Kramers-Kronig relations link the real and the imaginary parts of the complex modulus such that the causality condition is verified:

$$G'(\omega) = G_\infty + \frac{2}{\pi}\int_0^\infty \frac{uG''(u)}{\omega^2-u^2}\mathrm{d}u \tag{1a}$$

$$G''(\omega) = \frac{2\omega}{\pi}\int_0^\infty \frac{G'(u)}{u^2-\omega^2}\mathrm{d}u \tag{1b}$$

In [2], this method is applied only to DMA measurements of the shear modulus. In this work, it is applied independently to both shear and bulk moduli so that the built master curves fulfil the best causality conditions.
The results presented in Figure 3 at a chosen reference temperature of $12^o$C show that the frequency dependency of the shear modulus is much more pronounced than for the bulk modulus. A fractional derivative model [3] is identified for both moduli to describe their frequency-dependency (straight lines

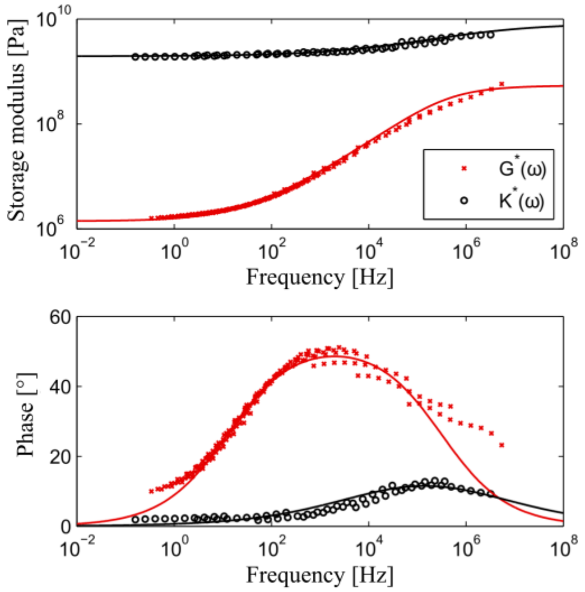


Figure 1: Dynamic shear and bulk moduli at $12^oC$.

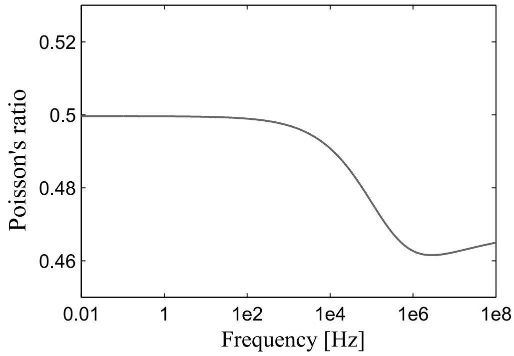


Figure 2: Real part of the Poisson's ratio determined indirectly from shear and bulk moduli measurements.

in Figure 3):

$$\begin{aligned} G^*(\omega) &= \frac{G_0 + G_\infty (i\omega\tau_G)^{\alpha_G}}{1+(i\omega\tau_G)^{\alpha_G}} \\ K^*(\omega) &= \frac{K_0 + K_\infty (i\omega\tau_K)^{\alpha_K}}{1+(i\omega\tau_K)^{\alpha_K}} \end{aligned} \tag{2}$$

where $G_\infty = G^*(\omega \to \infty) = G(t \to 0)$ and $K_\infty = K^*(\omega \to \infty) = K(t \to 0)$ are unrelaxed moduli, $G_0 = G^*(\omega \to 0) = G(t \to \infty)$ and $K_0 = K^*(\omega \to 0) = K(t \to \infty)$ are relaxed moduli, $\tau_G$ and $\tau_K$ are relaxation times, $\alpha_G$ and $\alpha_K$ represent the order of the fractional derivative in the constitutive equations in the time domain. The parameters of the identified viscoelastic models are :

$$\begin{aligned} &G_0 = 1.4 \text{ MPa}, \quad G_\infty = 0.54 \text{ GPa}, \quad \tau_G = 0.52\ \mu\text{s}, \quad \alpha_G = 0.59 \\ &K_0 = 1.95 \text{ GPa}, \quad K_\infty = 8.3 \text{ GPa}, \quad \tau_K = 0.15\ \mu\text{s}, \quad \alpha_K = 0.36 \end{aligned} \tag{3}$$

The identified viscoelastic models of the shear and the bulk moduli allow for the indirect determination of the Poisson's ratio. Figure 3 shows that it is very close to 0.5 which is consistent with the assumption of quasi-incompressibility of rubbers. It can also be observed that the Poisson's ratio remains constant up to about 1kHz, and then decreases up to 1MHz (measurements are not sufficient enough to extrapolate above 1MHz). These results agree well with those presented in [1] and obtained using different characterisation techniques.
From this study, it can be concluded that the assumption of a constant bulk modulus or Poisson's ratio should be carefully considered. In the case of Deltane 350, this assumption is valid only on a limited frequency range.

## References

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