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A Measurement Setup for the Determination of Temperature-Dependent Viscoelastic Material Parameters Using an Ultrasonic Pulse-Echo Technique

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Abstract: Ultrasonic methods facilitate a non-destructive characterisation of mechanical properties, such as Young's modulus or shear modulus, based on the propagation time and attenuation of ultrasonic signals. This contribution presents an ultrasonic measurement setup for determining temperature-dependent viscoelastic material parameters of polymers within a frequency range of $0.75\,\mathrm{MHz}$ to $2.5\,\mathrm{MHz}$. The system is based on a pulse-echo technique and features a temperature-controlled specimen chamber that enables precise and reproducible heating of hollow cylindrical specimens. To span the frequency range, a broadband ultrasonic transducer and a burst signal with a variable centre frequency are used for transmission. By systematically varying the temperature while measuring ultrasonic time-of-flight and attenuation, elastic and viscous properties can be identified across a temperature range of $20\,^{\circ}\mathrm{C}$ to $55\,^{\circ}\mathrm{C}$. Initial values are estimated from the measured signals, and the resulting viscous material parameters are determined using an inverse approach. The proposed method thus offers a time-efficient, non-invasive approach for characterising thermally sensitive, frequency-dependent material parameters.

Keywords: viscoelastic material parameters, temperature-dependent, inverse approach, pulse-echo measurement, polymers

Motivation

The determination of temperature-dependent viscoelastic material parameters is of crucial importance for understanding the behaviour of polymers in dynamic or harsh environments. Design, simulation and testing must be particularly accurate when polymers are subjected to continuous stress. However, the material parameters provided by manufacturers are often insufficient for simulation purposes, as they are typically measured using quasi-static and destructive methods. To address this limitation, acoustic methods have emerged as a viable alternative for non-destructive measurement [1, 2].

Our recent studies described the potential of an ultrasonic pulse-echo measurement setup and a guided wave approach for characterising viscoelastic properties of hollow cylindrical polymer specimens [3, 4]. The proposed framework, an inverse procedure, is presented in our work [5], where we demonstrate rapid convergence. The present study extends this methodology by systematically varying the ambient temperature between $20\,^{\circ}\mathrm{C}$ and $55\,^{\circ}\mathrm{C}$ of a specimen during measurements. Changing the thermal state perturbs a polymer's viscoelastic parameters e.g., elastic moduli, yet the specimen geometry remains (mostly) the same. Therefore, each temperature step results in a distinct pulse-echo measurement from the same physical specimen. By applying our inverse algorithm to this set of measurements, we can verify that the determined parameters evolve consistently with the imposed temperature changes, thereby demonstrating the robustness and repeatability of the method under varying operational conditions.

Forward Model of a Specimen with Viscoelastic Material Parameters

The forward model of the specimen is briefly introduced in the following. A hollow cylinder of inner radius r_i, outer radius r_o and length l is considered. Due to the extrusion process during the fabrication of polymer rods, a transversely isotropic viscoelastic material behaviour is assumed. The properties along the plane perpendicular to the cylinder length axis (indices $1, 2$) are assumed isotropic, while the properties along the longitudinal direction (index 3) differ. The polymer is assumed to behave linearly elastic for small strains and the constitutive relationship between the

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strains $\boldsymbol{\varepsilon}$ and the stresses $\boldsymbol{\sigma}$ is given by Hooke's law:

$$\boldsymbol{\sigma} = \mathbf{D}\,\boldsymbol{\varepsilon} \tag{1}$$

Considering transverse isotropy, the elasticity matrix $\mathbf{D}$ contains only five independent entries and can be expressed in terms of the five elastic parameters $E_1, E_3, \nu_{12}, \nu_{13}, \mu_{13}$, the Young's moduli, the Poisson's ratios and the shear modulus, respectively [3].

Further, a finite-element discretisation of the hollow cylinder gives the semi-discrete dynamic equilibrium

$$\mathbf{M}\,\ddot{\boldsymbol{u}}(t) \;+\; \mathbf{C}\,\dot{\boldsymbol{u}}(t) \;+\; \mathbf{K}\,\boldsymbol{u}(t) \;=\; \mathbf{0}\,, \tag{2}$$

where $\boldsymbol{u}(t)$ is the nodal displacement vector, $\mathbf{M}$ the consistent mass matrix and $\mathbf{K}$ the stiffness matrix built from the elasticity matrix as $\mathbf{K} = \int_{\Omega} \mathbf{B}^{\top}\mathbf{D}\mathbf{B}\,\mathrm{d}\Omega$. In this study, $\mathbf{C}$ denotes the damping matrix, which is modelled using the Rayleigh damping law

$$\mathbf{C} = \alpha_{\mathrm{M}}\,\mathbf{M} \;+\; \alpha_{\mathrm{K}}\,\mathbf{K}\,, \tag{3}$$

where α_{M} (mass-proportional) and α_{K} (stiffness-proportional) are scalar coefficients. Substituting the stiffness matrix and the Rayleigh damping (3) into the equation of motion (2) yields a governing relation for the hollow-cylindrical, transversely-isotropic polymer specimen subjected to a prescribed displacement. This relation can be solved with any standard time-integration scheme to obtain the displacement, velocity and stress fields needed for further analysis or for inverse property identification. The vector of all relevant viscoelastic parameters is given by

$$\boldsymbol{\gamma} = [E_1,\, E_3,\, \nu_{12},\, \nu_{13},\, \mu_{13},\, \alpha_{\mathrm{M}},\, \alpha_{\mathrm{K}}]\,. \tag{4}$$

Predicting ultrasonic wave propagation under viscoelastic constitutive laws can be computationally demanding, particularly in 3D finite element analyses at high frequencies and during parameter estimation in inverse procedures. To address this, the semi-analytical scaled boundary finite element method (SBFEM) is employed for efficient computation [6]. This method leverages a combination of radial discretisation, analytical solutions along the axis, and Fourier decomposition in the circumferential direction, thus efficiently handling arbitrary excitation patterns and the cylindrical geometry. The SBFEM formulation further enables the computation of sensitivities, i.e., derivatives of the simulated output with respect to the viscoelastic parameters $\boldsymbol{\gamma}$ [7]. Consequently, a single forward simulation results in both the time-dependent displacement response and its sensitivity to the underlying viscoelastic parameters, facilitating gradient-based optimisation.

So far, the descriptions refer only to modelling of the specimen. To set up a full forward model, the hardware of the measurement setup must also be modelled. For this purpose, analytical models are employed, which provide a swift yet sufficient representation of the hardware and are further introduced in [3].

Measurement Setup

Fig. 1 depicts the measurement setup including a USB oscilloscope, two custom-designed current-feedback amplifiers, switching electronics, a broadband ultrasonic transducer, and a thermal chamber with integrated radiator. Besides two inputs for data acquisition, the USB oscilloscope also features an arbitrary signal generator and an additional interface to trigger the switching electronics. The broadband amplifiers provide precise signal amplification of $6\,\mathrm{dB}$ when transmitting and $46\,\mathrm{dB}$ when receiving. The

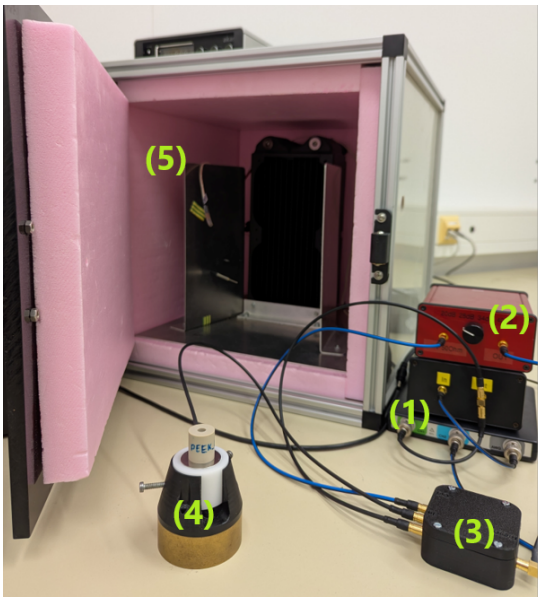

Fig. 1: (1) shows the USB oscilloscope (HS5-540XM, TiePie), (2) the custom transmitting and receiving voltage amplifier, (3) is the switching electronics with low on resistance, (4) is a broadband ultrasonic transducer with a specimen above, (5) is the thermal chamber with radiator.

wide bandwidth of the self-manufactured transducers allows excitation of variable center frequencies from $0.75\,\mathrm{MHz}$ to $2.5\,\mathrm{MHz}$, enabling the study of frequency effects on material parameters without the need to re-couple the specimen. Further, the specimen is coupled using a viscous medium applied by screen printing. The thermal chamber shown in Fig. 1 is connected to a cooling circulator (Julabo MAGIO MS-310F), which ensures fast and stable regulation of the temperature inside the chamber in a range from $20\,^{\circ}\mathrm{C}$ to $55\,^{\circ}\mathrm{C}$. In addition, the experimental setup is located in a temperature-controlled room

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that is maintained at $21 \pm 1\,^{\circ}\mathrm{C}$ to minimize external influences on the electronics.

Three different polymers are considered, with all specimens having the same dimensions. Polyetheretherketone (PEEK), polyamide 6 (PA6) and polypropylene (PP) are investigated. The specimens', inner radius $r_\mathrm{i} = 3\,\mathrm{mm}$, the outer radius $r_\mathrm{o} = 9\,\mathrm{mm}$ and length $l = 20\,\mathrm{mm}$ are considered.

Inverse Approach

This work employs an inverse approach for estimating the elastic material parameters of hollow-cylindric polymers in the ultrasound regime at different temperatures. A more detailed introduction to the method can be found in [5] and is briefly presented here. The method formulates material parameter identification as a nonlinear least-square-type optimisation problem, seeking to minimise the deviation between simulation and measurement by adjusting the model parameters $\hat{\boldsymbol{\gamma}}$. Leveraging a modification of the Levenberg-Marquardt method, based on geometric insights of the least-squares objective, this approach enhances convergence rates and reduces the required number of forward model evaluations. A key innovation is the objective function based on the autocorrelation of the signal's envelope in the frequency domain and can be written as:

$$\boldsymbol{\gamma} = \arg\min_{\hat{\boldsymbol{\gamma}}} \frac{1}{2} \left\| \arg_{\mathrm{stable}}(a) - \arg_{\mathrm{stable}}(\hat{a}(\hat{\boldsymbol{\gamma}})) \right\|^2$$

where a and $\hat{a}(\hat{\boldsymbol{\gamma}})$ are the autocorrelations of the measured and simulated envelopes, respectively. Further, $\arg_{\mathrm{stable}}(\cdot)$ denotes the stable, unwrapped phase extraction with damping for numerical stability. This transformation leads to a more convex optimisation landscape, effectively mitigating local minima and increasing the robustness and reliability of parameter estimation.

The initial estimates of the elastic parameters of $\boldsymbol{\gamma}$ are obtained from the group velocities of longitudinal and transverse sound $c_{\mathrm{g,L}}$ and $c_{\mathrm{g,T}}$, respectively (see, e.g., [3]). These are obtained from cross-correlation of the transmitted and received signals. The initial Rayleigh damping parameters are estimated in the frequency domain by evaluating the measured damping ratios α_{att} across a range of centre frequencies f_c to determine the corresponding α_M and α_K values:

$$\frac{\alpha_M}{4\pi f_\mathrm{c}} + \alpha_K \pi f_\mathrm{c} = \frac{\alpha_{\mathrm{att}}(2\pi f_\mathrm{c}) c_{\mathrm{g,L}}(2\pi f_\mathrm{c})}{2\pi f_\mathrm{c}} \,.$$

Results and Conclusion

Fig. 2 and Fig. 3 show the measured group velocities from the temperature and frequency dependent measurements of PEEK and PA6. While $c_{\mathrm{g,L}}$ increases with frequency, conversely it decreases with increasing heat. However, the selected temperature range and the heating of PA6 (same for PP) result in difficulties when determining $c_{\mathrm{g,T}}$. Similar observations are made in [8, 9] for PA6 and other polymers, discussing the glass transition temperature as the cause and subsequently restricting the temperature range significantly. Near the glass-transition temperature, the polymer's elastic moduli and viscosity change rapidly, causing strong dispersion and a large increase in ultrasonic attenuation that invalidates the simple linear-elastic assumptions used for parameter identification. Consequently the propagating wave is highly damped and scattered. As such, the transmitted signal is too distorted to extract reliable sound velocity or attenuation data. In order to provide only reliable estimates of the parameters, the temperature range from $20\,^{\circ}\mathrm{C}$ to $40\,^{\circ}\mathrm{C}$ is evaluated in the follow-

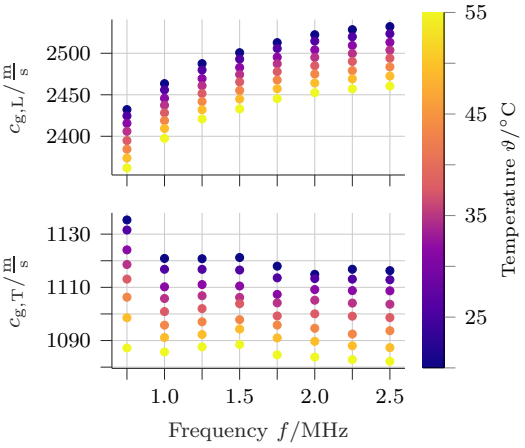

Fig. 2: Measured group sound velocities of PEEK.

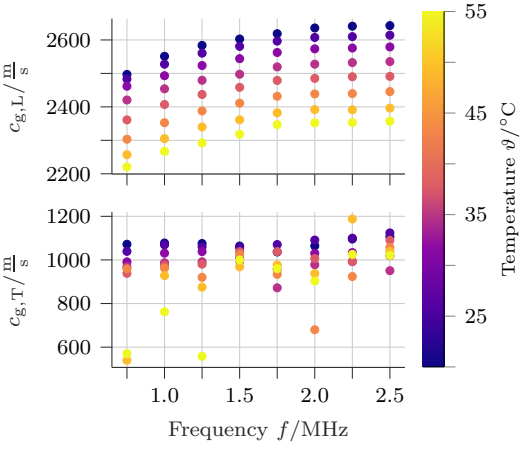

Fig. 3: Measured group sound velocities of PA6.

Tab. 1: Estimated material parameters of PEEK, PA6 and PP at temperatures ϑ.

Parameters	$\vartheta/°\mathrm{C}$				
PEEK	20	25	30	35	40
E_1/GPa	4.43	4.38	4.36	4.31	4.24
E_3/GPa	4.70	4.71	4.60	4.57	4.56
ν_{12}	0.37	0.37	0.37	0.37	0.37
ν_{13}	0.38	0.38	0.38	0.38	0.38
μ_{13}/GPa	1.57	1.56	1.55	1.54	1.53
$\alpha_\mathrm{M}/10^3$	127.2	124.0	130.2	130.6	131.1
$\alpha_\mathrm{K}/10^{-9}$	0.35	0.38	0.41	0.42	0.45

Parameters	$\vartheta/°\mathrm{C}$				
PA6	20	25	30	35	40
E_1/GPa	3.80	3.66	3.55	3.54	3.02
E_3/GPa	3.97	3.78	3.62	3.62	3.25
ν_{12}	0.39	0.39	0.40	0.41	0.40
ν_{13}	0.40	0.40	0.40	0.39	0.42
μ_{13}/GPa	1.26	1.19	1.08	0.96	0.99
$\alpha_\mathrm{M}/10^3$	157.5	174.5	182.8	195.9	217.1
$\alpha_\mathrm{K}/10^{-9}$	1.52	1.49	1.65	2.04	2.12

Parameters	$\vartheta/°\mathrm{C}$				
PP	20	25	30	35	40
E_1/GPa	3.87	3.43	3.44	3.41	3.21
E_3/GPa	4.03	4.08	3.40	3.42	3.16
ν_{12}	0.32	0.32	0.35	0.33	0.34
ν_{13}	0.33	0.34	0.34	0.33	0.34
μ_{13}/GPa	1.41	1.24	1.32	1.27	1.22
$\alpha_\mathrm{M}/10^3$	310.4	322.3	354.8	376.3	358.7
$\alpha_\mathrm{K}/10^{-9}$	1.93	1.97	2.00	2.04	2.01

ing. Tab. 1 shows the parameters of the materials determined at $f_\mathrm{c} = 1\,\mathrm{MHz}$ using the inverse method. Heating the test specimen to shift the operating point and test the robustness of the algorithm has been successfully implemented, particularly for PEEK. The identified parameters change approximately linearly with temperature. It is reasonable to conclude that the identified parameters are plausible, given the change in the respective sound velocities. For instance, an increase in temperature is associated with a decrease in the measured transversal sound velocity, whilst the identified shear modulus also decreases. PA6 and PP differ more at higher temperatures, as measured sound velocities differ by hundreds of metres per second, see Fig. 3. This indicates that no minor alteration in the operating point has been attained in this instance, and the parameter shift must be comparatively high. The results and the trend of the parameters presented is comparable qualitatively with the results of [8, 9]. In our case, transverse isotropy is assumed, but there is good agreement with the literature regarding the change in sound velocities. The consideration of other temperature-stable polymers can further illustrate the robustness of the measurement method and is the goal for further research.

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References

[1] L. Claes et al. "Determination of the material properties of polymers using laser-generated broadband ultrasound". In: *Journal of Sensors and Sensor Systems* 5.1 (2016), pp. 187–196. DOI: 10.5194/jsss-5-187-2016.

[2] M. Held et al. "Determination of Isotropic Elastic Constants from Dispersion Images Based on Ultrasonic Guided Waves by Using Neural Networks". In: *Ultrasonics* 143 (July 2024), p. 107403. DOI: 10.1016/j.ultras.2024.107403.

[3] D. Dreiling et al. "A pulse-echo measurement setup to determine viscoelastic material parameters". In: vol. 2822. 1. 2024. DOI: 10.1088/1742-6596/2822/1/012169.

[4] D. Dreiling et al. "Increasing the Sensitivity of Ultrasonic Transmission Measurements for Elastic Material Parameter Estimation". In: *Measurement Science and Technology* (May 2025). DOI: 10.1088/1361-6501/add9b6.

[5] D. Itner et al. "A modified Levenberg-Marquardt method for estimating the elastic material parameters of polymer waveguides using residuals between autocorrelated frequency responses". In: *arXiv* (2025). DOI: 10.48550/arXiv.2507.01706.

[6] D. Itner et al. "Efficient semi-analytical simulation of elastic guided waves in cylinders subject to arbitrary non-symmetric loads". In: *Ultrasonics* 114 (2021). DOI: https://doi.org/10.1016/j.ultras.2021.106389.

[7] M. S. Chowdhury, C. Song, and W. Gao. "Shape sensitivity analysis of stress intensity factors by the scaled boundary finite element method". In: *Engineering Fracture Mechanics* 116 (2014), pp. 13–30. DOI: 10.1016/j.engfracmech.2013.12.007.

[8] N. Lagakos et al. "Frequency and temperature dependence of elastic moduli of polymers". In: *Journal of Applied Physics* 59.12 (June 1986), pp. 4017–4031. DOI: 10.1063/1.336707.

[9] M. Webersen. "Zerstörungsfreie Charakterisierung der elastischen Materialeigenschaften thermoplastischer Polymerwerkstoffe mittels Ultraschall". PhD thesis. Universität Paderborn, 2021. DOI: 10.17619/UNIPB/1-1088.