Dynamic Torsion Test for the Mechanical Characterization of Soft Biological Tissues

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presented by
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Davide Valtorta
Zurich, January 2007
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Abstract

In this thesis, a novel measurement method for the characterization of the mechanical properties of soft biological tissues is presented. The linear viscoelastic properties are determined through dynamic torsion tests by applying forced torsional oscillations to soft tissue samples. This work presents the definition of the measurement principle, with the design of torsional resonating sensors and the development of analytical and finite elements methods used for the inverse material characterization. The reliability and limitations of the proposed measurement technique have been assessed with experiments on soft biological materials as well as with synthetic materials.

The viscoelastic response of soft materials is characterized for harmonic shear deformations at high frequencies (1-12 kHz) and small strains (up to 0.2% nominal strain for the soft biological tissues considered). Experiments are performed using a torsional resonating sensor, hereafter referred to as the torsional resonator device (TRD), which consists of a rod excited to vibrate at resonance, with one end in contact with a material sample. The resonating sensor induces shear waves in the material analyzed. Adherence between vibrating sensor and material sample is ensured by vacuum clamping in the contact area. The response of the material results in changes in the dynamic behavior of the vibrating system sensor + material sample. The damping characteristics and resonance frequency of the vibrating system are inferred from the control variables of a phase stabilization loop. These quantities are then related to the mechanical properties of the material using analytical and finite element models that describe the interaction between sensor and material sample.

In this work, soft biological tissues are assumed to be homogenous, isotropic materials with a linear viscoelastic response. This assumption can be considered suitable to describe the mechanical behavior of bulky internal organs, such as liver or kidney, with no or limited reinforcement by muscular fibers. By controlling the vibration amplitude of the sensor to be within the linear viscoelastic limits, soft tissues can be characterized by a complex shear modulus $G^*$, or equivalently by its storage and loss moduli. Due to the characteristics of the sensors proposed in this work, which have the capability to operate at several different torsional eigenfrequencies, the frequency dependent behavior of the complex shear modulus can be investigated.
The elastodynamic problem of forced torsional oscillations exerted on a viscoelastic medium, which describes the interaction between the vibrating sensor and the material samples, is solved using analytical and finite element models. The solution of this problem is presented for a variety of contact configurations and samples geometries. The measurement technique can be applied to test samples of undefined geometry, to samples of well defined and finite dimensions, to layered materials and thin membrane-like samples.

The measurement technique was validated through comparative measurements on synthetic material samples with wave propagation methods. Results obtained in tests executed ex vivo on bovine soft internal organs such as liver, kidney and uterus are presented in this work, discussing the possible error sources and uncertainties of the measurement. The measurement method was also applied to the characterization of synthetic materials such as bituminous binders, silicones and rubbers, electroactive polymers, and this demonstrates the versatility of this high frequency rheometry technique.

The torsional resonator device (TRD) presented in this work can be considered a useful tool for a fast and non-destructive characterization of soft biological materials and can lead to future applications of this technique for in vivo tests for diagnostics purposes.
In questa tesi viene presentata una nuova tecnica di misura per la caratterizzazione delle proprietà meccaniche nei tessuti biologici soffici. Le proprietà viscoelastiche di questi materiali sono determinate mediante un metodo di misura dinamico basato sull’esecuzione di oscillazioni torsionali forzate su campioni di tessuti molli. Questo studio si occupa della definizione del principio di misura, del disegno e della realizzazione dei sensori, dello sviluppo di soluzioni analitiche e numeriche che consentono di ottenere una caratterizzazione meccanica di campioni di materiali soffici. L’affidabilità e le limitazioni della tecnica di misura proposta sono state valutate attraverso esperimenti eseguiti sia su tessuti biologici che su materiali sintetici caratterizzati da basse rigidezze come gomme, materiali polimerici e leganti bituminosi.

La risposta viscoelastica dei tessuti biologici è caratterizzata attraverso oscillazioni armoniche in torsione eseguite ad alta frequenza (1-12 kHz) nel campo delle piccole deformazioni (fino a valori dello 0.2% per i tessuti molli considerati). Gli esperimenti sono stati eseguiti usando un sensore basato sul metodo della risonanza meccanica, detto torsional resonator device (TRD). Questo sensore consiste in una barretta che viene eccitata a vibrare attorno al proprio asse con una frequenza vicina ai modi propri torsionali, operando quindi in condizione di risonanza. L’estremità inferiore del sensore viene posta in contatto con un campione di materiale, nel quale vengono indotte onde di taglio dovute al movimento oscillatorio del sensore. La presenza del campione di materiale posto a contatto con il sensore si traduce in un cambiamento del comportamento dinamico del sistema meccanico complessivo, formato da sensore e campione di materiale. La frequenza di risonanza del sistema vibrante e la sua caratteristica di smorzamento vengono determinate grazie ad un sistema di controllo elettronico interamente basato sulla misura della fase relativa tra segnale in ingresso ed uscita, che permette la caratterizzazione dinamica del sistema. Dalla misura di queste due quantità dinamiche, le proprietà meccaniche del materiale analizzato sono ricavate attraverso modelli analitici e numerici (FEM) che descrivono l’interazione tra sensore vibrante e campione di materiale.

In questo lavoro, i tessuti biologici soffici vengono studiati assumendo l’ipotesi di materiale omogeneo ed isotropo. Questa ipotesi restrittiva può essere comunque considerata valida nel descrivere il comportamento meccanico di organi interni molli, come fegato o reni, nei quali la ridotta presenza di fibre muscolari ne limita il comportamento anisotropo. L’ampiezza di vibrazione del sensore TRD viene controllata...
in modo da restare entro i limiti della viscoelasticità lineare, permettendo una caratterizzazione dei tessuti biologici molli in termini del modulo di taglio complesso $G^*$, o equivalentemente mediante i moduli di accumulo elastico e di perdita. La capacità del sensore TRD di operare a diverse frequenze, corrispondenti alle frequenze proprie torsionali, permette di studiare il comportamento del modulo di taglio complesso al variare della frequenza e quindi a diverse velocità di deformazione.

L’interazione tra sensore vibrante e campione di materiale analizzato si riduce allo studio di un problema elastodinamico, dove vibrazioni forzate torsionali sono indotte in un mezzo viscoelastico. L’analisi di questo problema viene effettuata servendosi di soluzioni analitiche e soluzioni basate sul metodo degli elementi finiti, considerando una serie di possibili configurazioni di contatto e diverse geometrie del campione di materiale analizzato. In questo modo, questa nuova tecnica di misura può essere applicata per caratterizzare campioni di materiale dalla geometria indefinita, campioni di geometria ben assegnata e dimensioni finite, campioni di materiali non omogenei (stratificati) e membrane sottili.

La tecnica di misura oggetto di questo studio è stata validata mediante misure comparative effettuate su campioni di materiali sintetici per i quali sono stati impiegati metodi standard di test basati sulla propagazione delle onde meccaniche. Questa dissertazione raccoglie i risultati ottenuti in misure eseguite in vitro su campioni di organi interni di fegato, reni ed utero estratti da bovini. Il modulo di taglio complesso in funzione della frequenza viene riportato discutendo le fonti di errore e le incertezza di misura, confrontando i risultati con metodi di misura analoghi sviluppati per il test delle proprietà meccaniche dei materiali biologici. Il metodo di misura basato sulla vibrazione indotte da un risonatore torsionale è stato inoltre applicato alla caratterizzazione di materiali sintetici come gomme e siliconi, bitumi e leganti bituminosi, polimeri elettroattivi. In queste diverse applicazioni vengono dimostrate capacità e versatilità di questa tecnica reologica di misura ad alta frequenza.

Il torsional resonator device (TRD) sviluppato in questa ricerca può essere considerato uno strumento utile per una veloce esecuzione di misure non distruttive su tessuti biologici sottili e suggerisce la possibilità di future applicazioni in vivo, in camera operatoria, per una caratterizzazione meccanica a fini diagnostici.
## List of Symbols

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Unit</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_m$</td>
<td>[-]</td>
<td>Coefficient of the displacement of a downgoing wave in the $m^{th}$ layer</td>
</tr>
<tr>
<td>$B_m$</td>
<td>[-]</td>
<td>Coefficient of the displacement of an upgoing wave in the $m^{th}$ layer</td>
</tr>
<tr>
<td>$C_{n0}$</td>
<td>[Pa]</td>
<td>Coefficient of the strain energy function $W$</td>
</tr>
<tr>
<td>$D$</td>
<td>[J]</td>
<td>Energy loss in a vibration period</td>
</tr>
<tr>
<td>$E^*$</td>
<td>[Pa]</td>
<td>Complex Young’s modulus</td>
</tr>
<tr>
<td>$E_0$</td>
<td>[Pa]</td>
<td>Instantaneous Young’s modulus</td>
</tr>
<tr>
<td>$E_1$</td>
<td>[Pa]</td>
<td>Storage Young’s modulus</td>
</tr>
<tr>
<td>$E_2$</td>
<td>[Pa]</td>
<td>Loss Young’s modulus</td>
</tr>
<tr>
<td>$G^*$</td>
<td>[Pa]</td>
<td>Complex shear modulus</td>
</tr>
<tr>
<td>$G_0$</td>
<td>[Pa]</td>
<td>Instantaneous shear modulus</td>
</tr>
<tr>
<td>$G_1$</td>
<td>[Pa]</td>
<td>Storage shear modulus</td>
</tr>
<tr>
<td>$G_2$</td>
<td>[Pa]</td>
<td>Loss shear modulus</td>
</tr>
<tr>
<td>$H_1$</td>
<td>[-]</td>
<td>Hankel transform of order 1</td>
</tr>
<tr>
<td>$I$</td>
<td>[-]</td>
<td>Equivalent stiffness coefficient</td>
</tr>
<tr>
<td>$J_1$</td>
<td>[-]</td>
<td>Bessel function of the first kind of order 1</td>
</tr>
<tr>
<td>$J_p$</td>
<td>[m$^4$]</td>
<td>Polar moment of inertia</td>
</tr>
<tr>
<td>$L$</td>
<td>[m]</td>
<td>Length</td>
</tr>
<tr>
<td>$M$</td>
<td>[N·m]</td>
<td>Torque</td>
</tr>
<tr>
<td>$Q$</td>
<td>[-]</td>
<td>Quality factor</td>
</tr>
<tr>
<td>$R$</td>
<td>[-]</td>
<td>Equivalent damping coefficient</td>
</tr>
<tr>
<td>Symbol</td>
<td>Unit</td>
<td>Description</td>
</tr>
<tr>
<td>--------</td>
<td>------</td>
<td>-------------</td>
</tr>
<tr>
<td>$T$</td>
<td>[$^\circ$C]</td>
<td>Temperature</td>
</tr>
<tr>
<td>$TF$</td>
<td>[-]</td>
<td>Transfer function of a vibrating system</td>
</tr>
<tr>
<td>$U_M$</td>
<td>[J]</td>
<td>Maximum potential energy of a vibrating system</td>
</tr>
<tr>
<td>$W$</td>
<td>[Pa]</td>
<td>Strain energy function</td>
</tr>
<tr>
<td>$Z_T$</td>
<td>[N·s·m$^{-1}$]</td>
<td>Torsional mechanical impedance</td>
</tr>
<tr>
<td>$a$</td>
<td>[m]</td>
<td>Radius of a torsional vibrator</td>
</tr>
<tr>
<td>$c$</td>
<td>[N·m]</td>
<td>Stiffness coefficient of an equivalent torsional spring</td>
</tr>
<tr>
<td>$c_s$</td>
<td>[m·s$^{-1}$]</td>
<td>Shear wave velocity in a sensor</td>
</tr>
<tr>
<td>$c_{SH}$</td>
<td>[m·s$^{-1}$]</td>
<td>Shear wave velocity in a viscoelastic medium</td>
</tr>
<tr>
<td>$d$</td>
<td>[m]</td>
<td>Diameter</td>
</tr>
<tr>
<td>$d_f$</td>
<td>[N·m·s]</td>
<td>Damping coefficient of an equivalent torsional spring</td>
</tr>
<tr>
<td>$df$</td>
<td>[Hz]</td>
<td>Damping characteristic</td>
</tr>
<tr>
<td>$f$</td>
<td>[Hz]</td>
<td>Frequency</td>
</tr>
<tr>
<td>$f_{res}$</td>
<td>[Hz]</td>
<td>Resonance frequency of a vibrating system</td>
</tr>
<tr>
<td>$f^+$</td>
<td>[Hz]</td>
<td>Frequency corresponding to $\Delta \phi = +\frac{\pi}{4}$ with respect to resonance</td>
</tr>
<tr>
<td>$f^-$</td>
<td>[Hz]</td>
<td>Frequency corresponding to $\Delta \phi = -\frac{\pi}{4}$ with respect to resonance</td>
</tr>
<tr>
<td>$\tilde{g}(\xi,z)$</td>
<td>[-]</td>
<td>Hankel transform of the $a$ function $g$</td>
</tr>
<tr>
<td>$h$</td>
<td>[m]</td>
<td>Height of the portion of a vibrating rod embedded into a medium</td>
</tr>
<tr>
<td>$h_m$</td>
<td>[m]</td>
<td>Thickness of the $m^{th}$ layer</td>
</tr>
<tr>
<td>$j$</td>
<td>[-]</td>
<td>Square root of $-1$</td>
</tr>
<tr>
<td>$k_0$</td>
<td>[-]</td>
<td>Dimensionless wave number in a viscoelastic medium</td>
</tr>
<tr>
<td>$k_s$</td>
<td>[-]</td>
<td>Wave number in shear in a sensor</td>
</tr>
<tr>
<td>$p$</td>
<td>[Pa]</td>
<td>Pressure</td>
</tr>
<tr>
<td>$q$</td>
<td>[m]</td>
<td>Thickness of a membrane-like sample</td>
</tr>
<tr>
<td>$r$</td>
<td>[m]</td>
<td>Radial distance in a cylindrical coordinate system</td>
</tr>
<tr>
<td>$s$</td>
<td>[-]</td>
<td>Laplace transform variable</td>
</tr>
<tr>
<td>$t$</td>
<td>[s]</td>
<td>Time</td>
</tr>
</tbody>
</table>
\( \mathbf{u} \) [m] Displacement vector

\( u_i \) [m] Scalar components of the displacement vector in the \( i \)-direction

\( z \) [m] Vertical distance in a cylindrical coordinate system

\( \alpha \) [rad] Phase shift angle in the phase-locked loop

\( \beta_m \) [-] \( \beta_m = \sqrt{\xi_0^2 - k_0^2} \)

\( \gamma \) [-] Engineering shear strain

\( \delta \) [rad] Phase angle of the complex shear modulus \( G^* \)

\( \theta \) [rad] Amplitude of a torsional vibration

\( \lambda \) [-] Stretch ratio

\( \lambda_{SH} \) [m] Wavelength of a SH-wave

\( \mu \) [Pa·s] Viscosity of a fluid

\( \nu \) [-] Poisson’s number

\( \xi \) [-] Hankel transform variable

\( \rho \) [kg·m\(^{-3}\)] Density of a medium

\( \tau_{ij} \) [Pa] Shear stress, subscripts indicate the components of the stress tensor

\( \phi \) [rad] Phase angle of the transfer function \( TF \) of a vibrating system

\( \phi_T \) [rad] Phase angle of the complex torsional mechanical impedance \( Z_T \)

\( \varphi \) [rad] Azimuth in a cylindrical coordinate system

\( \chi_{max} \) [m] Maximum element size in a FE model

\( \omega \) [rad·s\(^{-1}\)] Angular frequency

\( \overset{\cdot}{\quad} \) Derivative with respect to time

\( \overset{\cdot}{\quad},i \) Derivative with respect to a variable \( i \)

\( \hat{\quad} \) Indicates a complex amplitude

\( \vec{\quad} \) Indicates a unit vector

Literature references are indicated by [ ] brackets.

Equations are indicated by ( ) brackets.
Chapter 1

Introduction

Accurate characterization of the mechanical behavior of biological tissues is essential to a number of new computer-aided medical technologies, such as surgical training, planning and development of real-time surgical simulators [6, 14, 69, 86]. Furthermore, mechanical modeling of soft tissues is needed in the development of new surgical tools and can contribute to diagnostics, distinguishing healthy and unhealthy tissues depending on their mechanical response.

In our society, the increasing demand of high-fidelity surgical tools for training, planning, and intra-operative support, has lead researchers to focus their attention on biomechanics, in various aspects of modeling the mechanical behavior of biological tissues. The Swiss National Science Foundation has started in 2001 to fund a major research project, called CO-ME (COnputer aided and image guided MEdical interventions), that involved a network of scientists and engineers with the common task to develop and evaluate basic and applied technologies, required for diagnosis, surgical planning and therapeutic intervention. This work is a part of the CO-ME Project 6, which received the assignment to develop constitutive models to describe the mechanical behavior of soft biological tissues and to create new experimental techniques for the assessment of their mechanical properties.

A new measurement technique for biological tissues has been developed aiming at: (i) obtaining a non-destructive test, suitable for in vivo application during open surgery, and (ii) complementing the existing quasi-static tissue characterization already existing at the Institute of Mechanical Systems, ETH Zurich, namely the Aspiration Device [60, 91].
Experimental techniques for testing biological tissues

Characterizing the mechanical behavior that soft nonload-bearing tissues exhibit is a difficult task. Standard methods for testing soft tissues are needed to produce repeatable results that can be mathematically interpreted in order to describe the natural behavior of the tissue. Mechanical testing of biosolids can be accomplished with different methodologies, mainly grouped into destructive and non-destructive techniques [20, 22, 90].

Destructive testing utilizes material samples directly extracted from organs and experiments are carried out according to standard methods of material characterization, such as tensile, compression and shear tests. In the works of Yamada [94] and Fung [22], a large number of experiments on all kinds of human and animal tissues are described. Among all the methods reported in their works, uniaxial and biaxial tensile tests appear often and are carried out with standard equipment for material tests. One major issue that has to be faced in testing biological tissues with standard machines is however represented by samples preparation and handling [55]. Realizing a precise excision from the tissues analyzed of samples of well defined geometry is a common problem of all these methods. Compression tests are used as alternative to test those samples, whose characteristics do not allow clamping in tensile tests, for example with soft organs such as liver, kidneys, brain or uterus (see e.g. [11, 19, 56, 92]). However, the release of fluids that occurs due to compression of the samples can highly influence the outcomes of these tests. Shear tests have also been carried out using standard rheometers on excised tissues samples (see e.g. [16, 47, 59]). The use of conventional shear rheometers, normally employed in dynamic mechanical analysis of fluids and polymers, enables the viscoelastic characteristics of biological tissues to be investigated up to frequency limits of 10 to 100 Hz.

Non-destructive techniques, in contrast to the methods presented above, present the great advantage of a possible direct application in vivo, during open surgery, avoiding the problems of samples preparation and handling and eliminating the uncertainties due to the alterations of the material when extracted from its biochemical environment [39]. Most of these techniques are based on indentation tests, in which a mechanical indentor probe is pushed against the surface of a tissue sample and the response of the tissue is recorded in terms of force and displacement in time, as in the works of Ottensmeyer [65] and Zheng [100, 101]. An alternative to indentation tests is offered by the so called pipette aspiration technique [3], which sets well defined boundary conditions through the contact of an aspiration tube with a soft tissue sample and yields two dimensional load-deformation data. The aspiration technique was applied during in vivo measurements on soft human organs by Kauer et al. [37] and recently by Nava et al. [61].

Major research efforts have also been made to use non-invasive imaging methods such as ultrasound and magnetic resonance for the evaluation of soft tissue elas-
ticity parameters [23, 27]. These methods, which combine mechanical deformation and measurement of resulting strain fields to extract elasticity data, allow a characterization of tissue regions that are not directly accessible with standard testing methods. Among these imaging techniques, elastography [64, 78] and sonoelasticity [41, 45] can be employed in the detection of regional stiffening within one organ. In magnetic resonance elastography (MRE) [58], quantitative measurement of the elastic properties is realized by monitoring the wave propagation field induced in a tissue by an external mechanical vibrator (see e.g. [42, 51, 82]).

The dynamic torsion test

This work aimed at developing a new non-destructive testing method for soft tissues based on the know-how gained in the last twenty years at the Institute of Mechanical Systems, ETH Zurich, on torsional vibrating sensors. Dynamic testing, instead of quasi-static, is performed by making a material sample part of a vibrating system and achieves a material characterization in the frequency domain, giving useful information about the strain rate dependence of the mechanical properties of the samples analyzed.

Sensors based on forced torsional oscillations and dynamic shear tests are widely employed in research and industrial processes in several application fields. A good review of this measurement technique and of the design of torsional resonating probes is reported by Langdon [43]. The Institute of Mechanical Systems, ETH Zurich, has developed several sensors based on this working principle and applied them in the rheological characterization of fluids and suspensions at high frequencies with the works of Dual, Goodbread and Sayir [18, 79] and more recently by Häusler, Hochuli and Romoscanu [29, 31, 75]. In their works, they presented torsional resonating probes vibrating at high frequencies (always above 1 kHz) to measure the viscoelastic properties in shear of viscous fluids [18], polymers and suspensions [75], bitumen [31], blood [29]. High frequency rheometry is useful to characterize the viscoelastic properties of polymers and fluids and can give information about their microstructure [21] by observing their typical frequency dependent behavior. The study of the material response at different frequencies, and thus different wavelengths, allow investigating the influence of the tissue microstructure on the mechanical behavior of soft biological tissues.

In this framework, a new non-destructive technique to measure the mechanical properties of soft tissues was developed: the dynamic torsion test. The mechanical response of viscoelastic materials is characterized for harmonic shear deformations performed at high frequencies (1-12 kHz) and small strains (up to 0.2% nominal strain for the soft biological tissues considered). Experiments are performed using a torsional resonating sensor, hereafter referred to as the torsional resonator device (TRD), which consists of a rod excited to vibrate at resonance, with one end in contact with a material sample. The TRD induces shear waves in the material analyzed.
1. Introduction

through forced torsional oscillations: the viscoelastic properties of the material are derived from the results of the interaction between the sensor and the material sample considered. This technique complements the Aspiration Device [60] and allows obtaining a characterization of soft tissues in a wide range of strains and loading rates.

Outline of the present work

In this thesis, a novel experimental method for the mechanical characterization of soft biological tissues was developed. Several aspects has to be faced in the development of a new measurement technique:

(i) the sensors design, with features that must be tuned to the application considered here: in particular, the experiment must provide well defined kinematic and static boundary conditions,

(ii) the mechanical modeling of the interaction between sensors and soft tissues, that is crucial for the quantitative evaluation of the experimental results,

(iii) the analysis of the scatter in experimental results, error sources and uncertainties in the measurements.

The dynamic torsion tests was thought specifically for biomechanics and resulted in the design of a sensor, the torsional resonator device (TRD), that is able to characterize soft biological tissues. Nonetheless, this measurement technique can be applied to different applications fields, exploiting always the same working principle. Collaboration with the EMPA, Swiss Federal Laboratories for Material Testing and Research, has showed how this measurement technique can be effective in the mechanical characterization of synthetic material as silicone rubbers, bituminous binders, and electroactive elastomers.

The measurement principle of the dynamic torsion test is described in Chapter 2. Details on the experimental technique are reported, along with the description of the design of the devices used and the mechanical models that describe their dynamic behavior. The sensors presented in this work are able to measure the torsional mechanical impedance of the material samples analyzed, that will be then link to their viscoelastic properties.

The torsional mechanical impedance measured with a sensor can be related to the rheological properties of the material analyzed by using a model that accurately describe the interaction between torsional vibrating sensor and material sample. In Chapter 3, the elastodynamic problem of forced torsional oscillations that describe the interaction is solved, using analytical and finite element approaches. A discussion about the suitability of the dynamic torsion test for different application fields is also
included, highlighting the most important factors that must be controlled during a test and the inherent error sources.

Chapter 4 deals with the characterization of soft biological tissues with the torsional resonator device. The measurement technique is first validated with comparative wave propagation experiments on silicone phantoms that mimic the mechanical properties of soft tissues. Then, results obtained ex vivo on bovine internal organs are presented. The viscoelastic behavior of liver, kidney and uterus is characterized at high frequencies. Discussion on the application of this method in biomechanics is provided, along with an analysis of the error sources and possible improvements of the method.

The dynamic torsion test, developed for the main purpose of characterizing soft biological tissues, is applied in Chapter 5 to synthetic material. Here, the results of a collaboration with the EMPA are showed (i) in studying the aging process of bituminous binders, that influences the efficiency of road pavements, and (ii) for the mechanical characterization of electroactive polymers, showing the effectiveness of this measurement method in different application fields.

Finally, the achievements of the present works are summarized in Chapter 6.
1. Introduction
Chapter 2

The Dynamic Torsion Test

This chapter describes the measurement principle of the dynamic torsion test. In this test, forced torsional oscillations are exerted by a vibrating sensor on a material sample in order to characterize its viscoelastic behavior. Details on the measurement principle of this technique are reported, along with the description of the design of the devices used and the mechanical models that describe their dynamic behavior. The execution of a dynamic torsion test provides the measurement of a mechanical quantity, the torsional impedance, that will be linked in Chapter 3 to the material properties of the medium analyzed.

2.1 Introduction

The mechanical properties of viscoelastic materials are determined in this work using a dynamic test. In a dynamic test, the material sample under investigation is made part of a vibrating system (usually a shaker or a resonating sensor) and the material properties are extracted by measuring the change in the dynamic behavior of the system. In this thesis, the sensors analyzed are excited to vibrate at their torsional natural frequencies. This test is based on the principles of the so-called resonance methods [10, 28, 68] widely used in parameters identification and structural mechanics: here, the structure under analysis consists of a vibrating sensor, whose geometry and properties are given, and a material sample of unknown properties.

Use of oscillating probes for the characterization of the rheological properties has been proposed for fluid films [18, 43, 54, 80] and suspensions [75]. These sensors consist of tubes or pipes excited to vibrate in torsional resonance and interacting with a viscoelastic material. The know-how in the application of this technique at the Institute of Mechanical Systems, ETH Zurich, dates back to the late 80s with the work of Sayir, Dual and Goodbread [18, 79]. This technique was applied to the
viscometry of fluids, later extended to the rheology of suspensions [75], bituminous binders [31], and recently in biomechanics with a blood viscosimeter developed by Klaus Häusler [29].

This thesis deals with the development of a similar measurement method based on forced torsional oscillations, where sensors are excited to vibrate at their eigenfrequencies. These sensors consists of rod-shaped vibrators, whose lower extremity is laid on the top of a material sample. In contrast to existing devices, the sensors presented in this work interact with the material samples only through the contact area at the lower extremity of the rod-shaped sensors. In some cases, the contact area can also include the sides of the sensors. The mechanical properties are derived from the material response to harmonic shear in the linear viscoelasticity range at high frequencies (1-12 kHz).

The proposed measurement method has been used for different applications, always exploiting the same principle: (i) in biomechanics, for the determination of the viscoelastic properties of soft biological tissues; (ii) for the characterization of synthetic materials, as silicone rubbers and polymers; (iii) in bitumen rheology, to monitor the evolution of the viscoelastic properties due to exposure to environmental influences. The specific conditions and working environments typical of these various application fields require the development of a different design for the devices employed, whose features will be described in this chapter.

In Section 2.2, the working principle of this class of torsional resonating sensors is presented, along with the control electronics used to measure the changes in their dynamic behavior. The design of the vibrating sensors used in this thesis are presented in Section 2.3. These are: (i) the torsional resonator device (TRD) developed for soft biological tissues and (ii) the high frequency torsional rheometer (HFTR), used in viscometry of fluids and for the characterization of synthetic materials. A crucial point in the development of a measurement technique based on dynamic testing is represented by the mechanical modeling of the sensors. Their dynamic behavior must be accurately modeled and the interaction with the viscoelastic medium is characterized in terms of a quantity, the torsional mechanical impedance, that will be used (as described in Chapter 3) to extract the material parameters of the material samples.

### 2.2 Measurement principle

Figure 2.1 shows a simplified scheme of the dynamic torsion test. The torsional vibrator consists of a cylinder made of metal (i.e. steel, aluminum or brass, of known material properties $G^*_{s}$ and $\rho_{s}$) driven in torsional oscillations by an electromagnetic transducer. The transducer applies a torque $M_e(t) = \hat{M}_e e^{j2\pi ft}$, a time harmonic function with frequency $f$, with $\hat{M}_e$ indicating the amplitude of the torque in complex notation. The rotation of the sensor around its axis is described by the angle $\theta(x,t) = \hat{\theta}(x)e^{j2\pi ft}$ that is also a harmonic function.
The lower extremity of the sensor is in contact with a material sample that is assumed to behave as a homogenous, linear viscoelastic material of unknown properties $G^*$ and $\rho$. This assumption, that considerably simplifies the problem, will be discussed in detail in the next chapters, depending on the materials and testing conditions considered.

The frequency of excitation $f$ is chosen to be close to one of the torsional eigenfrequencies of the mechanical system (vibrator+viscoelastic medium). The sensor is indeed a resonator, i.e. a device that vibrates around its eigenfrequencies. The techniques available for parameters identification of a mechanical system at resonance (resonance methods [10, 28, 68]) can be employed.

An important issue in resonance testing is represented by the choice of geometry, design and material of the torsional resonator that determine the eigenfrequencies of the system. The sensors considered in this thesis have resonance frequencies in the range 1-12 kHz: therefore, the viscoelastic material samples will be tested in this frequency domain. The use of a resonance method implies a technique that is not spectroscopic, i.e. a full frequency characterization of the materials is not allowed: they will be characterized only in correspondence to the eigenfrequencies of the sensor chosen (discrete characterization in the frequency domain).

![Figure 2.1: The dynamic torsion test: scheme of the torsional resonator interacting with a viscoelastic medium.](image)
2. The Dynamic Torsion Test

![Figure 2.2: Transfer functions of the vibrating system. Comparison between a calibration run (sensor vibrating in air) and a measurement run (sensor in contact with a material sample).]

### 2.2.1 Description of the mechanical behavior at resonance

The measurement technique used by the sensors presented in this work relies entirely on the principle that any interaction with a viscoelastic medium will change the dynamic behavior of the torsional resonator. The behavior of the vibrating mechanical system can be described with its complex transfer function $\hat{T}\hat{F}(f)$ (function of the frequency $f$) as the ratio between the resulting motion of the vibrator $\theta(x_0, t)$ in a reference position $x_0$ and the excitation torque $M_e(t)$. Choosing for simplicity the reference point $x_0$ at the tip of the resonator, in correspondence to the contact with the material sample, $\theta(x_0, t) = \theta_0(t) = \dot{\theta}_0 e^{j2\pi ft}$ and follows that:

$$\hat{T}\hat{F}(f) = \frac{\theta(x_0, t)}{M_e(t)} = \frac{\dot{\theta}_0}{M_e} \tag{2.1}$$

Significant changes in the dynamic behavior of the vibrating system occur as the free end of the resonator adheres to a material sample. Figure 2.2 shows the typical transfer function of the system, vibrating at resonance during a calibration run (measurement executed without contact with the material) and a measurement run (placing the free end in contact with the material sample). Two parameters, which can be evaluated from the phase curve ($\phi = \text{Arg}(\hat{T}\hat{F})$), characterize the dynamic behavior of the system: the resonance frequency $f_{\text{res}}$, and the quality factor $Q [10]$. The quality factor is defined in Equation 2.2 and is proportional to the ratio of maximum potential energy stored in the vibrating structure $U_M$ and the energy loss.
2.2. Measurement principle

\[ D \text{ due to damping in one vibration period:} \]

\[ Q = \frac{2\pi U_M}{D} \tag{2.2} \]

\( Q \) can be determined from the phase curve in the vicinity of resonance \([9, 10]\), as in Equation 2.3:

\[ Q = \frac{f_{res}}{df} \tag{2.3} \]

\[ df = f^+ \left( \phi = \frac{\pi}{2} + \frac{\pi}{4} \right) - f^- \left( \phi = \frac{\pi}{2} - \frac{\pi}{4} \right) \tag{2.4} \]

where the two measured frequencies \( f^+ \) and \( f^- \) (see Figure 2.2) correspond to a phase shift difference \( \Delta \phi = \pm \pi/4 \) with respect to the resonance frequency \( f_{res} \) (where \( \phi = \pi/2 \)). The difference between the resonance frequency measured in a calibration run in air and a measurement with a viscoelastic medium in contact is referred to as the resonance frequency shift \( \Delta f_{res} \), as indicated in Figure 2.2.

2.2.2 Phase stabilization loop

The resonance frequency \( f_{res} \) and the damping characteristic \( df \) are obtained experimentally using a method initially developed by Joseph Goodbread at the Institute of Mechanical Systems, ETH Zurich. The method is outlined in Figure 2.3; a full description of the electronic circuit is given in Sayir et al. [79]. Basically, a periodic square voltage signal (frequency \( f \)) is produced by a voltage controlled power amplified function generator (VCO). This signal is simultaneously fed to a coil, which electromagnetically drives the resonator with an excitation torque \( M_e \), as well as to a phase shifter (PS), which alternatively shifts the signal by a fixed angle \( \phi_S = \pi/2 \pm \Delta \alpha \). The motion of the mechanical oscillator is picked up by a second electromagnetic transducer, yielding a signal with the same frequency \( f \) and a phase shift \( \phi_M \). Both the electrically and mechanically phase-shifted signals are fed to a phase sensitive detector that returns the phase difference \( \Delta \phi \). This phase difference is used by a PI controller that transforms the signal into a tension increment \( \delta V \) that is used to correct the frequency of the VCO function generator. This results in a phase-locked loop, where the driving frequency \( f \) is continuously adjusted until sensed and PS-shifted signals are in phase \( (\Delta \phi = 0) \), and the PI correcting tension vanishes.

By choosing the phase shifter angle \( \Delta \alpha = 0 \), the resonance frequency of the mechanical system \( f_{res} \) is searched for (the difference between the input signals and signal indicating the motion of the mechanical system equals \( \pi/2 \)). The choice of \( \Delta \alpha = \pm \pi/4 \) allows determining the two frequencies \( f^+ \) and \( f^- \) defined in Equation 2.4. The difference \( df \) and the average of the driving frequencies corresponding to both phase shifts \( \pi/2 \pm \pi/4 \) are computed. Their average value over a given period
of time, usually set to 1 second, is recorded as the resonance frequency $f_{res}$ of the system, while their difference represents the damping characteristic $df$. The control loop allows stabilizing the system so to ensure high accuracy ($10^{-6} \cdot f_{res}$ Hz) in the determination of $f_{res}$ and $df$ [79]. No amplitude measurement is involved in the control loop.

The method described above is a stationary method that implies the use of two electromagnetic transducers, one for driving the motion and one for sensing it. The same control logic can be applied to sensors with only one transducer, acting as a driver of the torsional motion, and at the same time as an encoder of the motion [79], alternating cycles of excitation and measurement. Considering a period of $n_{tot}$ working cycles at the same frequency $f$, after a given number of driving cycles $n_D$, the transducer turns into a sensor for $n_{tot} - n_D$ cycles (so-called gating). The number of working cycles is set to $n_{tot} = 15$ and $n_D$ can vary between 3 and 8. However, this arrangement implies that the measurement is not performed under strictly stationary conditions, since driving periods are periodically interrupted by sensing ones, where the system is in actual free decay. The advantages of this method are a reduction in the complexity of the device, since only one electromagnetic transducer is required, and the elimination of any cross-talk between actuator and sensor transducers. In the following, this method will be referred to as quasi-stationary. Romoscanu [74] presented in his thesis a detailed analysis of the quasi-stationary method, showing the precision of this method as a function of the sensing cycles $n_D$ and the quality factor of the resonating sensor. The quasi-stationary method delivers precise results when the $Q$ factor of the mechanical system is higher than 1000 (that is always the case with measurements of soft biological tissues, as it will be shown in Chapter 4).
2.2. Measurement principle

With lower $Q$ factors, the motion of the resonator during the sensing phase will be sensibly reduced by the higher damping. Therefore, the number of sensing cycles $n_{tot} - n_D$ must be reduced as much possible to prevent this problem [74].

In this thesis, both arrangements will be employed; the sensor designed for soft biological tissue measurements (the TRD introduced in Section 2.3.1) will use a stationary measurement method, while the quasi-stationary method will be employed for other applications with synthetic material (HFTR sensor described in Section 2.3.2).

2.2.3 Modeling the dynamic behavior of the sensors

In the dynamic torsion test, the influence of a viscoelastic material on the vibrating sensor is found during the tests with the measurement of $\Delta f_{res}$ and $df$. This information can be directly used to quantitatively distinguish materials of different properties, without physical interpretation of the result. In order to extract the material parameters of the medium in contact, a step further is required: an accurate mechanical model of the sensor, capable of traducing the two measured values $\Delta f_{res}$ and $df$ into a physical quantity as a torque or an impedance.

Figure 2.4 presents a simplified mechanical scheme of the sensors used for the dynamic torsion test. The sensor is modeled as a continuous, deformable beam characterized by given parameters as the shear modulus $G^*_s$, the density $\rho_s$, radius $a$ of the circular cross-section, length $L$ and polar moment of inertia $J_p$. The behavior of the beam is described by the classical wave equation in one dimension, with
The Dynamic Torsion Test

harmonic solution ($\omega = 2\pi f$ is the angular frequency):

$$\theta_{xx} + k_s^2 \ddot{\theta} = 0 \quad (2.5)$$

$$k_s^2 = \frac{\omega^2}{c_s^2} = \frac{\omega^2}{G_s \rho_s} \quad (2.6)$$

where $c_s$ is the shear wave speed and $k_s$ the wave number in the beam. The harmonic excitation torque $M_e(t) = \hat{M}_e e^{j\omega t}$ situated along the beam drives the system into torsional oscillations. The rotation angle in correspondence to the contact with the viscoelastic medium is indicated as $\theta_0$ and will also be a time harmonic function:

$$\theta_0(t) = \hat{\theta}_0 e^{j\omega t} \quad (2.7)$$

The effect of the viscoelastic medium on the sensor is introduced in terms of a torque $M_v$ acting on it:

$$M_v(t) = \hat{M}_v e^{j\omega t} \quad (2.8)$$

The relation between the complex amplitude of the viscoelastic torque $\hat{M}_v$ and the amplitude of the torsional vibration applied by the sensor on the medium $\hat{\theta}_0$ can be written as:

$$\hat{M}_v = (c + j d) \hat{\theta}_0 \quad (2.9)$$

where the torque $\hat{M}_v$ is substituted by an equivalent torsional spring-dashpot element, $c$ and $d$ being its stiffness and damping constants, respectively. Alternatively, another quantity can be used to describe this interaction, the torsional mechanical impedance $Z_T$, defined in Equation 2.10 by dividing the torque acting on the vibrator by the angular velocity of the sensor $\hat{\theta}_0$ and the contact area:

$$Z_T = \frac{\hat{M}_v}{\hat{\theta}_0} \cdot \frac{1}{\pi a^2} = \frac{\hat{M}_v}{j \omega \hat{\theta}_0} \cdot \frac{1}{\pi a^2} \quad (2.10)$$

Considering now the mechanical system depicted in Figure 2.4, the concentrated excitation torque $M_e$ divides the beam into two domains ($L_1$ and $L_2$). Within each domain, the behavior is described by the rotation angle $\theta_i(x_i, t)$ with $i = 1, 2$, general solution of Equation 2.5:

$$\theta_i(x_i, t) = \hat{\theta}_i(x_i) e^{j\omega t} = (a_i \sin k_s x_i + b_i \cos k_s x_i) e^{j\omega t} \quad (2.11)$$

where the four parameters $a_i, b_i$ for $i = 1, 2$ must be solved by imposing boundary
2.3 Sensors Design

and equilibrium equations:

\[ \theta_1(0, t) = 0 \]  \hspace{2cm} (2.12)

\[ \theta_1(L_1, t) = \theta_2(0, t) \] \hspace{2cm} (2.13)

\[ G_s J_p \theta_{1,x}(L_1, t) = G_s^* J_p \theta_{2,x}(0, t) + M_e \] \hspace{2cm} (2.14)

\[ G_s J_p \theta_{2,x}(L_2, t) = -M_v \] \hspace{2cm} (2.15)

\[ M_v = -Z_T \pi a^2 \dot{\theta}_2(L_2, t) \] \hspace{2cm} (2.16)

Once the coefficients \( a_i, b_i \) have been determined, the transfer function of the system is found as:

\[ \overline{TF}(\omega) = \frac{\theta_2(L_2, t)}{M_e(t)} = \overline{TF}(\omega, G_s^*, \rho_s, a, J_p, L_1, L_2) \] \hspace{2cm} (2.17)

that is a function of the angular frequency of excitation, of the material and geometric properties of the sensor, and of the torsional mechanical impedance of the viscoelastic medium in contact \( Z_T \). The complete solution is not reported here for space reason.

With the analytical model of the sensor described above, the experimental values of \( \Delta f_{res} \) and \( df \) obtained in a test with a viscoelastic medium can be converted to the torsional mechanical impedance \( Z_T \). To a specified value of \( Z_T \) corresponds a specific transfer function \( \overline{TF}(\omega, Z_T) \), from which the values of \( \Delta f_{res} \) and \( df \) can be evaluated at each resonance frequency. In Appendix A.1 and A.2, further details are given and more accurate models for the sensors used in this thesis are presented. The sensors described in this thesis can provide accurate measurements of the torsional mechanical impedance of a medium. In Chapter 3, it will be shown how from a measurement of \( Z_T \) is possible to extract the rheological parameter \( G^* \) of the viscoelastic medium analyzed. Finite element models are also necessary to treat special cases where the sensors or the material samples have particular geometries that can only be modeled with a numerical approach. In Chapter 3, more details about the possibilities given by finite element models in describing the behavior of these sensors and their interaction with other materials will be presented.

2.3 Sensors Design

In the previous section, the working principle of the class of sensors based on forced torsional oscillations has been presented. This measurement principle can be applied to different sensors design, depending on the particular application and materials that have to be analyzed. In this thesis, two different devices will be considered:

- (i) the Torsional Resonator Device (TRD) designed for testing soft biological tissues,
- (ii) the High Frequency Torsional Rheometer (HFTR) designed for viscosity
measurements of fluids and suspensions and used in this work to determine the rheological properties of asphalt mixtures and bituminous binders.

The design details as well as the mechanical models used to describe the dynamic behavior of these sensors are presented here. The sensor described in this thesis were conceived with and manufactured by Dr. Klaus Häusler at the Institute of Mechanical Systems, ETH Zurich.

2.3.1 The Torsional Resonator Device

The Torsional Resonator Device (TRD) was designed to test the mechanical properties of soft biological tissues. The purpose of this device is to obtain local and accurate measurements of the mechanical properties of soft tissues, typically internal organs as liver, kidney, uterus. Soft tissues are characterized in the high frequency region (1-12 kHz), corresponding to the first five eigenfrequencies of the TRD probe.

Several issues had to be considered in the design phase of this device, as:

- the high sensitivity required in testing soft biological tissues, presenting values of $|G^*|$ in the range 2-500 kPa,
- the necessity to perform measurements in a short period of time to avoid tissues dehydration, which strongly affects their mechanical properties,
- the difficulty to formulate the boundary conditions that defines the interaction between vibrating sensor and soft tissues. Perfect adherence between sensor and material must be ensured to prevent any sliding that could not be correctly modeled and would lead to inaccurate measurements,
- the necessity to develop a device that could be used in the future in the operation room for in vivo tests, with the capability of miniaturization for use in laparoscopic surgery.

Figure 2.5 shows the TRD in the bench-top configuration. The device is fixed to a stand and can be moved vertically using a travel stage to touch the tissue sample, which lays on a balance to ensure that no axial force is exerted on the tissue (resolution $10^{-1}$ mN). The control box on the right of Figure 2.5 contains the electronics and is used to excite the torsional motion and to measure the two quantities, resonance frequency $f_{res}$ and damping characteristic $df$, that will be later used for the material parameters extraction. The logic of the control box relies on the phase-locked loop technique with stationary measurement that was previously described in Section 2.2.2.

The sensor scheme is reported at the bottom of Figure 2.5. A tube made of brass is excited to torsional vibrations (around one of the first five torsional eigenfrequencies, in the range of 1-12 kHz) by an electromagnetic transducer, while a second electromagnetic transducer is used as sensor for measuring the motion. Two channels are connected to the control electronics, so that a continuous measurement is
2.3. Sensors Design

Figure 2.5: Torsional Resonator Device: photograph (above) and sketch (below).
2. The Dynamic Torsion Test

Figure 2.6: Scheme of the electromagnetic transducers used to excite or sense the torsional motion. Polarization of the permanent magnets indicated with N and S.

possible, allowing a higher precision of the measurement than the quasi-stationary one. The tube is clamped at one extremity with a decoupling mass. The other extremity is free for the so called ”calibration run”, and is in contact with the tissue sample for the ”measurement run”.

Each of the two electromagnetic transducers consists of a pair of permanent magnets, welded to the tube, and a pair of electric coils fixed to the external case of the sensor, with input current $i_e$. A sinusoidal current with frequency $f$ drives an oscillating magnetic field $B_e$ and induces a moment $M_e$ on the tube. If employed as a sensing transducer, the torsional motion of the tube modulates the magnetic field of the permanent magnets and induces a voltage in the electric coils. Figure 2.6 shows the scheme of the electromagnetic transducer used in this sensor to excite or sense the torsional motion of the TRD.

A detailed drawing of the TRD is reported in Figure 2.7, where the device is shown first assembled, and then with separated components after removal of the external coil-support case. The external case serves only as a support of the electric coils and is fixed to the decoupling mass through removable screws. Due to the higher inertia of the decoupling mass and of the external case, these two behave as an inertial frame for the system (see Appendix A.1 for details). Isolation of the system from the external environment is provided by a styrofoam or rubber insert of low stiffness (in this case, a rubber insert of 50 HB, Brinell hardness) glued between the decoupling mass and the aluminum support that serves as a stand. The core of the torsional resonator device is the tube, made of brass ($G_s^*$ and $\rho_s$ identify the shear modulus and density of the material used for the sensor), clamped at one extremity to a decoupling mass. The tube has a total length $L_{tot} = 388$ mm, an outer diameter $d_e = 6$ mm and inner diameter $d_i = 5.1$ mm. Two pairs of permanent magnets are welded to the tube in different positions and serve as excitation and sensing transducer, as indicated in Figure 2.7.
Figure 2.7: Torsional Resonator Device: construction scheme, geometric and material parameters.
Table 2.1: Geometric and material data of the TRD sensor.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_{tot}$</td>
<td>Total length of resonator</td>
<td>388 [mm]</td>
</tr>
<tr>
<td>$L_1$</td>
<td>Position of the excitation transducer</td>
<td>190 [mm]</td>
</tr>
<tr>
<td>$L_2$</td>
<td>Position of the sensing transducer</td>
<td>163 [mm]</td>
</tr>
<tr>
<td>$L_3$</td>
<td>Position of the contact disc</td>
<td>35 [mm]</td>
</tr>
<tr>
<td>$a$</td>
<td>Radius of the contact area on the disc</td>
<td>2.55 [mm]</td>
</tr>
<tr>
<td>$d_e$</td>
<td>External diameter of the brass tube</td>
<td>6 [mm]</td>
</tr>
<tr>
<td>$d_i$</td>
<td>Internal diameter of the brass tube</td>
<td>5.1 [mm]</td>
</tr>
<tr>
<td>$J_p$</td>
<td>Polar inertia moment of the tube</td>
<td>$6.08 \cdot 10^{-11}$ [kg·m$^4$]</td>
</tr>
<tr>
<td>$J_m$</td>
<td>Inertia moment of a magnet pair</td>
<td>$7.33 \cdot 10^{-9}$ [kg·m$^2$]</td>
</tr>
<tr>
<td>$J_d$</td>
<td>Inertia moment of the contact disc</td>
<td>$4.83 \cdot 10^{-10}$ [kg·m$^2$]</td>
</tr>
<tr>
<td>$J_{dec}$</td>
<td>Inertia moment of the decoupling mass</td>
<td>$4.9 \cdot 10^{-4}$ [kg·m$^2$]</td>
</tr>
<tr>
<td>$\rho_s$</td>
<td>Density of brass tube</td>
<td>8600 [kg/m$^3$]</td>
</tr>
<tr>
<td>$\rho_{dec}$</td>
<td>Density of stainless steel (decoupling mass)</td>
<td>8030 [kg/m$^3$]</td>
</tr>
<tr>
<td>$G_{1s}$</td>
<td>Elastic shear module of brass tube at 20°C</td>
<td>38.82 [GPa]</td>
</tr>
<tr>
<td>$Q_s$</td>
<td>Quality factor of brass (measured)</td>
<td>$\approx 20000$ [-]</td>
</tr>
</tbody>
</table>

At the other extremity of the tube, a disc with micro-openings is bonded. This disc has the very important function of ensuring adherence between the vibrating tube and the soft tissues in contact. The particular design of the disc mounted at the tip of the resonator is shown in Figure 2.7. The micro-openings have a width of 30 µm and are distributed uniformly on the circular area (radius $a = 2.55$ mm) where the contact with the material takes place. This particular arrangement is necessary to test soft biological tissues. Due to the typical viscosity of the surface of these tissues, in particular internal organs, sliding on the surface represents a potential source of significant uncertainties. It will be shown in Chapter 3 that, for the particular stress distribution in the contact region, sliding can take place at the borders of this region. Therefore, the openings must be as much close as possible to the external radius $a$ of the contact disc, as shown in the detail of Figure 2.7 (openings are designed up to a radius of $a - 30$ µm).

By evacuating the internal volume of the tube, adherence between resonator and soft tissues is obtained (vacuum clamping). A pressure reservoir and a pump are connected to the device through a flexible rubber hose. The pressure can be manually controlled by a valve. The pressure inside the tube must not be applied for long periods of time to avoid local dehydration of the tissue. After reducing the pressure inside the tube to a value of 0.2 bar for only 10 sec, contact is achieved with the tissue, that justify the assumption of perfect adherence (as demonstrated in Section 4.2). After this clamping phase, the measurement showed to be stable during ex-vivo experiments performed in the laboratory on soft tissue samples.

Due to the high precision (in the order of µm) required for the dimensions and openings of the contact disc, this has been manufactured from silicon wafers using
high precision microfabrication technologies. The disc can be replaced after usage if the openings are closed by blood or dust. Due to the small dimensions of the disc openings and the low underpressure applied in the tube (0.3 bar absolute pressure) no damage occurs in the tissue as a consequence of vacuum clamping. Absence of bleeding, lacerations and abrasions was verified by visual inspection after testing biological tissue samples. Table 2.1 reports the main geometric and material data of the TRD sensor depicted in Figure 2.7.

Using the geometric and material data of the sensor, an analytical model of the TRD can be derived (see Appendix A.1). The sensor is excited to vibrate in torsion around the first five torsional resonances of the system. The values of the first 5 resonances and mode shapes are reported in Figure 2.8. The second resonance frequency \( f^{(II)} = 3903 \text{ Hz} \) is not used in the measurements. Due to the presence of a bending mode at about 3880 Hz, a pure torsional motion of the sensor cannot be ensured, being the measurement influenced by the bending motion.

The TRD device exhibits a quality factor \( Q_s \approx 20000 \) (quality factor of the sensor vibrating in air) at each torsional frequency. The losses present in the system are the internal dissipation of the tube material (brass), the losses at the clamping position and due to the imperfections of the welded components. This high \( Q_s \) factor (compared to standard values of about 5000) is required for a good sensitivity of the device in testing soft tissues. Typically, in a measurement run with soft biological tissue, the quality factor \( Q \) is in the range of 3000. The difference between the two
quality factors is an index of the sensitivity of the device [74]. The vibration amplitude at the tip of the resonator is kept below 0.001 rad, limiting the shear strains to $\gamma_{\text{max}} < 0.2\%$, for the soft biological materials and range of frequencies considered in this thesis. At this strain amplitude, the tissue response is assumed to be linear viscoelastic, in agreement with the findings of [47] and [59].

A typical experimental procedure with TRD consists of the following steps: (i) a calibration run is performed; (ii) the resonator is put in contact with the material sample, and the internal pressure of the tube is decreased; (iii) a measurement run is performed. The whole procedure takes approximately 1 minute, and can be repeated for four torsional eigenfrequencies of the resonator. At the characteristic frequencies of these experiments, the observation time leads to several thousand oscillations periods, so that a steady harmonic response state is reached in the system.

Particular attention must be paid to any temperature difference between sensor and material analyzed. A difference of few degrees can already be critical, influencing the reliability of the results. The measure of the resonance frequency shift $\Delta f_{\text{res}}$ is affected by any temperature change of the sensor during the test. The material parameters of the sensor (in particular, the value of $G_{1s}$) are temperature dependent. A temperature decrease of $1^\circ \text{C}$ will traduce into an increase of resonance frequency of about 0.015%. At the first resonance frequency $f^{(1)} = 1300$ Hz, this corresponds to an increase of $\Delta f_{\text{res}}(\Delta T = 1^\circ \text{C}) \approx 0.2$ Hz, that is of the same order of magnitude of the $\Delta f_{\text{res}}(G^*)$ expected for contact with soft material samples ($|G^*| = 10^3 - 10^5 \text{ Pa}$). Especially in testing soft internal organs, this represents a potential source of uncertainties, as discussed in Chapter 4. The damping characteristics $d_f$, on the contrary, is not significantly affected by the temperature differences (maximum temperature range for the TRD $20 - 40^\circ \text{C}$), remaining $Q_s \approx 20000$.

### 2.3.2 The High Frequency Torsional Rheometer

The High Frequency Torsional Rheometer (HFTR) was originally designed to monitor the rheological properties of viscous fluids and suspensions by Dr. Klaus Häusler at the Institute of Mechanical Systems, ETH Zurich. This device is characterized by a more robust design than the previously described TRD and was originally thought to be used for in-line measurements of industrial processes for quality purposes, to monitor the viscosity of fluids.

Collaboration between the Department of Pavements and Sealing Components, EMPA, and the Institute of Mechanical Systems, ETH Zurich, was started in 1994 with application of this measurement technique in determining the mechanical properties of asphalt mixtures and bituminous binders. In the work of Hochuli [31], results on the rheology of bitumen were presented using a torsional rheometer based on forced torsional oscillations exerted in the high frequency domain. In [31], and previously in the characterization of fluids viscosity by Dual [18], the vibrating sensors were completely immersed in the material sample under analysis, the sides of the resonators being completely in contact with the material.
2.3. Sensors Design

Figure 2.9: High Frequency Torsional Rheometer (HFTR). Assembled device and components: external tube case, protective case, inner rod with permanent magnet.
In this thesis, the same type of rheometers are employed placing only their tip in contact with the samples, as in the case of the TRD. This type of contact, even though it reduces the sensitivity of the sensor by reducing the size of the interacting area, can be used to achieve local and surface characterization of solid materials. The work presented in this thesis concerning the rheological properties of bituminous binders is the result of a collaboration between the author of this thesis and Edoardo Mazza at the ETH Zurich, Lily Poulikakos and Manfred Partl at the EMPA.

The HFTR sensor is shown in Figure 2.9 with the whole assembled sensor and each of the components, along with a scheme of the device geometry and the material data. In contrast with the design of the TRD, a single electromagnetic transducer is present, covered by a protecting case that prevents any contact with the environment (thus enabling the use of the probe in aggressive environments). The external case is made of stainless steel (18-8 CrNi), while the inner rod can be made of different materials. The use of different materials for the inner rod changes the torsional operative frequency of the device. The sensor used in this thesis was built using a stainless steel inner rod (18-8 CrNi). The excitation is made on the top extremity of the internal rod. The electric coils are fixed to the flange as depicted in Figure 2.9 and a permanent magnet is fixed on the top extremity of the internal rod. The maximum vibration amplitude is observed in correspondence to the magnet, while no motion is observed at the root of the external case, as well as on the flange. The material data reported in Figure 2.9 were evaluated with wave propagation measurements or resonance methods in the laboratory, on the single components of the HFTR.

The device is designed to operate at one single torsional frequency, which is $f^{(I)} = 5090$ Hz at 20°C. An analytical model of the HFTR can be used to study its dynamic behavior (see Appendix A.2). Due to the specific design and assembly of this sensor (geometry difficult to be modeled with discrete or continous elements, presence of fillets, welded and brazed components), the finite element method was also employed to study its dynamic behavior. Figure 2.10 reports the first mode shapes of the sensor evaluated by a FE model developed using Abaqus (Hibbitt Karlsson and Sorenson Inc., Pawtucket, USA). The big flange at the top of the external tube acts as a decoupling mass for the vibrating system. Therefore, the protective case is not considered in this analysis. Two torsional frequencies are found in this analysis: $f^{(I)} = 5090$ Hz and $f^{(II)} = 18554$ Hz. Only the first one is chosen as operative frequency for measurements. The first eigenfrequency is far enough from other bending modes that could disturb the measure (the closest one is at $f_B^{(II)} = 4920$ Hz).

The HFTR is a robust device that can be used in environments where temperature and pressure vary during the test. A temperature sensor is placed inside the protective case to register any variation, enabling a temperature characterization of the material samples analyzed. However, the temperature influences also the dynamic behavior of the sensor itself by modifying the elastic properties and dimensions of the probe and must be taken into account when modeling its behavior. Figure 2.11 shows the results of calibration runs with the HFTR (i.e. measurements
Figure 2.10: Torsional resonance frequencies and mode shapes of the HFTR obtained from a finite element model developed in Abaqus. Results of a linear perturbation step to extract the eigenvalues of the model.

executed with the free sensor vibrating in air) in the temperature range 20-110°C, at ambient pressure. The resonance frequency decreases significantly with increasing temperatures, due to the decrease of the elastic constant $G_{ls}$. Analytical or finite element model must consider the variation of the elastic shear modulus of the sensor $G_{ls}$ with the temperature, which has the order of magnitude of a 3% variation each 100°C. The behavior observed in Figure 2.11 can be modeled considering a variation of the elastic shear modulus with the temperature $\frac{\Delta G_{ls}}{\Delta T} = -34 \text{ MPa/°C}$. The quality factor $Q_s$ of the resonating probe in air increases from a value of 3800 at 20°C to a value of 5700 at 110°C. In contrast to what was observed with the TRD (where $Q_s$ remains stable), the bigger temperature range covered by the HFTR requires the
variation of $G_{1,s}/G_{2,s} = Q_s(T)$ to be included in the mechanical model, following the trend registered in the experimental readings in air of Figure 2.11.

In this thesis, the contact with the materials analyzed with the HFTR lays on the lower tip of the sensor. A partial contact on the lateral sides of the sensor is also considered in the experiments presented in Chapter 5. No particular procedure was necessary to ensure adherence between sensor and material. The assumption of adherence is considered to be fulfilled to a wide extent in the case of the bituminous materials analyzed with the HFTR, which normally adhere easily to steel surfaces.

The HFTR for field tests

Another type of design of the HFTR is considered in this thesis for field tests on bituminous binders. Due to the robustness of the device in environment involving spurious low frequencies, typical of traffic-induced vibrations, the HFTR is a useful tool to monitor rheological changes directly on the field, on roads and pavements exposed to environmental conditions. A variant of the HFTR was designed specifically for field tests and used to monitor the rheological properties of an asphaltic plug joint situated on a highway bridge during three consecutive years of tests, from 1998 to 2000. The results recorded during the field tests are analyzed in this thesis (Chapter 5) in terms of the changes in the complex shear modulus $G^*$ due to the exposure to environmental conditions.

The HFTR for field tests was originally developed by Klaus Häusler and Andreas Hochuli at the Institute of Mechanical Systems, ETH Zurich, and was already employed during a first test campaign on bituminous binders in laboratory [31].
2.3. Sensors Design

Figure 2.12: High Frequency Torsional Rheometer for field tests. Scheme of the device (right) and device installed on a highway plug-joint (left).

Figure 2.12 shows the scheme of this device, which is very similar to the HFTR already described in the previous section. The device has an additional decoupling mass between the external tube and the protective case. Design details for this device are reported accurately in Hochuli’s thesis [31]. The second torsional frequency is the unique operative frequency used, \( f^{(II)} = 5318 \text{ Hz} \) with a value of \( Q_s = 2900 \) at \( 20^\circ \text{C} \). The stainless steel chosen for the construction of the device (18-8 CrNi) ensures the durability over the three years of tests (with exposure to rain, snow, salt, traffic-induced vibrations). The geometric properties of the HFTR for field tests are reported in Figure 2.13, while in Table 2.2 a list of the main material properties is summarized.

The control box was connected to a computer to record the data acquired on the field tests at intervals of 10 minutes, saving the information about time of the

<table>
<thead>
<tr>
<th>Variable</th>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>( a )</td>
<td>Radius of the contact area on the disc</td>
<td>6.75 [mm]</td>
</tr>
<tr>
<td>( G_{1s} )</td>
<td>Elastic shear module of stainless steel at 20°C</td>
<td>75.54 [GPa]</td>
</tr>
<tr>
<td>( \frac{\Delta G_{1s}}{\Delta T} )</td>
<td>Variation of elastic constant with temperature</td>
<td>-34 [MPa/°C]</td>
</tr>
<tr>
<td>( \rho_s )</td>
<td>Density of stainless steel</td>
<td>7900 [kg/m³]</td>
</tr>
<tr>
<td>( Q_s )</td>
<td>Quality factor of the sensor in air</td>
<td>2900 [-]</td>
</tr>
</tbody>
</table>

Table 2.2: Material data of the HFTR for field tests. Further details can be found in the work of Hochuli [31].
test (year, month, day, time), temperature $T$ of the test, resonance frequency $f_{\text{res}}$ and damping characteristic $df$. Prior to executing the experiments on the field, a series of calibration runs of the device in air have been recorded for a wide temperature range, obtaining a curve similar to the one reported in Figure 2.11 (see [31]). The value of $\Delta f_{\text{res}}$ was extracted by subtracting from the recorded values of resonance frequency obtained during the experiments on the plug joint the values of the resonance frequency obtained in air in the calibration.

In modeling the behavior of this sensor, there are no substantial differences with what was already presented for the standard HFTR in the previous section. An analytical model of the device can be used to predict its behavior and extract the torsional impedance from the measurements. Similarly, a finite element model of the sensor can be built to study the interaction between the sensor and the plug joint (see Chapter 3 and Chapter 5). The finite element approach presents in this case an advantage, since the contact condition with the plug joint (contact laying on the bottom of the contact disc and also on the lateral sides for a height of $h = 4$ mm) is difficult to be modeled analytically.
Chapter 3

Modeling the Interaction between Sensor and Material

The determination of the mechanical properties of viscoelastic materials through dynamic torsion tests requires an accurate modeling of the interaction between the torsional vibrating sensor and the material sample in contact. Once the torsional mechanical impedance of a material sample has been measured with a sensor, as discussed in Chapter 2, this quantity has to be linked to the rheological properties of the material by using a model that describe the interaction with it. The torsional impedance must be expressed as a function of the material parameters and frequency of excitation. The elastodynamic problem of forced torsional oscillations must be solved in terms of contact stresses and displacement field. This chapter deals with the description of the analytical and finite element models developed to this end and used for the inverse material characterization. A discussion about the suitability of the dynamic torsion test for different application fields is also included, highlighting the most important factors that must be controlled during a test and the possible error sources.

3.1 Introduction

Two possible ways exist for the determination of the mechanical properties of materials through dynamic torsion tests: (i) a calibration-based approach and (ii) an accurate modeling of the mechanical interaction between the probe and the material sample. In the first approach, the sensors must be calibrated with different material samples, whose mechanical properties are already known, in order to obtain reference values and compare them with the measurement readings. Calibration is complicated and needs a number of instrument constants [18, 43, 80] to be de-
3. Modeling the Interaction between Sensor and Material

determined before executing the tests, without providing a full comprehension of the measurement process. In the second approach, a better physical interpretation of the process is required, consisting in the determination of the contact stresses in the boundary region between vibrator and viscoelastic medium, in the calculation of the displacement field in the whole medium and finally in the determination of its torsional mechanical impedance. This quantity will be a function of the material parameters of the medium analyzed, of the size and geometry of the contact and of the frequency of excitation.

The physical approach to the problem offers more possibilities in investigating materials whose properties are not known a priori or difficult to be determined with alternative methods, and it is the approach discussed in this thesis. To this purpose, the complex mechanical shear waves (SH-waves) propagation problem in a viscoelastic medium, induced by the motion of the sensor, must be studied. The interaction between sensor and material can be reduced to a subject that has received considerable attention in the field of applied mechanics: the elastodynamic problem of a torsional vibrating source over a viscoelastic half-space (see Figure 3.1). Originally known as the Reissner-Sagoci problem [77], the SH-wave propagation generated by a rod-shaped vibrator in an elastic half-space has been solved analytically by many researchers [17, 50, 83], who were mainly interested in seismic investigations and foundations engineering. Robertson [73] was the first to suggest the possibility of extracting material parameters from torsional dynamic tests based on the variation of the resonance frequency of a vibrating source. The response of a rod vibrating source partially embedded in a half-space and subjected to torsional vibrations (see Figure 3.2) has also received some attention [4, 50, 57, 63, 72], leading to analytical and semi-analytical solutions.

The sensors described in Chapter 2 can simply be placed on the surface of a specimen or partially immersed into it up to a specified height. The specimen can be assumed to be homogenous or layered, i.e. characterized by different material parameters depending on the position. The shape and the size of the specimen analyzed can also change, affecting the impedance measurements. All these different testing configurations, depending on the boundary conditions and on the type of excitation applied to the medium, require the development of analytical and finite element solutions to treat problems ranging from the simple case of a rod-shaped vibrator over a half-space, to the case of a rod-shaped vibrator deeply embedded into it, to more complicated cases of a half-space consisting of multiple material layers and complicated contact geometries between vibrator and medium.

In this chapter, analytical and finite element solutions of the elastodynamic problem of forced torsional vibrations in a viscoelastic medium are presented. Analytical solutions represent a fast and reliable method to evaluate the interaction between the sensor and the medium in contact for simple cases and standard contact geometries. On the other side, finite element solutions are required to handle those cases where particular geometries or nonlinearities occur and no analytical approach is possible. Due to the numerical difficulties that often occur in solving this problem, the analytical solutions, obtained for simple cases, are used as a reference for valida-
3.2 Problem statement: forced torsional oscillations on a viscoelastic half-space

The typical interaction between the sensors presented in Chapter 2 and a material sample in contact is represented in Figure 3.1 and 3.2, where a torsional vibrating source placed on the surface or partially embedded in a viscoelastic half-space are depicted, respectively. The half-space assumption for the material sample in contact is reasonable when the dimensions of the sample are large enough to avoid any stationary effect resulting from waves reflected at the boundaries. This assumption will be further discussed in Section 3.3.3, where the effect of sample size is analyzed. The torsional vibrator, which exerts forced vibrations with angular frequency $\omega = 2\pi f$ on the medium, has radius $a$ and can also be embedded for a height $h$ into the medium, as shown in Figure 3.2. The material is characterized by the density $\rho$ and the complex shear modulus $G^*$, assuming linear viscoelasticity for small vibration amplitudes and describing its behavior in shear through the following equations:

$$\tau(t) = \left( G(0) + \int_0^\infty e^{-j\omega s} \hat{G}(s) ds \right) \cdot \gamma(t) = G^* \gamma(t)$$  \hspace{1cm} (3.1)

$$\gamma(t) = \gamma_0 e^{j\omega t}$$  \hspace{1cm} (3.2)

$$G^* = G_1 + jG_2 = |G^*| e^{j\delta}; \quad \delta = \arctan \frac{G_2}{G_1}$$  \hspace{1cm} (3.3)

**Figure 3.1:** Rigid rod-shaped vibrator placed on the top of a viscoelastic half-space.

**Figure 3.2:** Rigid rod-shaped vibrator embedded in a half-space for a height $h$. 
where \( \tau(t) \) and \( \gamma(t) \) represent the shear stress and engineering shear strain, respectively, and \( \omega \) is the angular frequency of their harmonic time function. The complex shear modulus \( G^* \) of the material can also be represented with its real and imaginary components \( G_1 \) and \( G_2 \), called respectively storage and loss shear modulus, or in the equivalent complex exponential notation, where \( |G^*| \) represents the magnitude and \( \delta \) the phase angle.

A cylindrical coordinate system \((r, \phi, z)\) is used to describe the half-space, with the unit vectors \( \vec{r}, \vec{\phi}, \vec{z} \) that identify the axis directions. The torsional vibrator is in contact with the surface of the half-space, vibrates around the \( z \)-axis and excites shear waves with displacement in the \( r-\phi \) plane (SH-waves) in the viscoelastic medium. Due to the kinematic boundary condition at the surface, the displacement vector field in the half-space can be described as in Equation 3.4, thus reducing to the azimuthal component \( u_\phi \) only:

\[
\begin{align*}
\mathbf{u} &= u_r \vec{r} + u_\phi \vec{\phi} + u_z \vec{z} = u_\phi (r, z, t) \vec{\phi} \quad u_r = u_z = 0
\end{align*}
\]

(3.4)

Considering from now on, for simplicity, the displacement \( u_\phi = u \) and assuming a time-harmonic displacement \( u(r, z, t) = u(r, z) e^{j\omega t} \), the equations of linear momentum and the kinematic relations lead here to the SH-waves Equation 3.5, where \( c_{SH} \) identifies the complex shear wave speed in the material of density \( \rho \):

\[
\frac{\partial^2 u}{\partial z^2} + \frac{\partial}{\partial r} \left[ \frac{1}{r} \frac{\partial (ru)}{\partial r} \right] + \frac{\omega^2}{c_{SH}^2} u = 0 \quad c_{SH}^2 = \frac{G^*}{\rho}
\]

(3.5)

**Boundary conditions**

The problem of a torsional vibrating source on a viscoelastic half-space, known as the Reissner-Sagoci problem [77] (see Figure 3.1), can be solved by specifying a set of boundary conditions, which can be either a stress distribution or a displacement field in the contact region of radius \( a \). In this work, a displacement boundary condition will always be prescribed at the interface with the viscoelastic medium, assuming perfect adherence with the sensor. Any uncertainty caused by sliding in the contact region must be avoided, as it was already mentioned in Chapter 2, with an appropriate design of the sensors. In case of adherence, the boundary conditions are:

\[
\begin{align*}
&u_\phi = \theta_0 r e^{j\omega t} \quad \text{for } 0 < r \leq a, \quad z = 0 \quad (3.6) \\
&\tau_{\phi z} = 0 \quad \text{for } r > a, \quad z = 0 \quad (3.7)
\end{align*}
\]

which identify a linear displacement field in the contact area of radius \( a \), where \( \theta_0 \) is the amplitude of the rotation applied by the vibrating source on the surface, and a stress-free surface outside the contact region.

Similarly, for the case depicted in Figure 3.2, where a part of the rigid rod-shaped vibrating source is embedded in the half-space, the boundary conditions can
be written as follows:

\[ u_\phi = \theta_0 ae^{j\omega t} \quad \text{for} \quad r = a, \quad 0 < z \leq h \quad (3.8) \]

\[ u_\phi = \theta_0 re^{j\omega t} \quad \text{for} \quad 0 < r \leq a, \quad z = h \quad (3.9) \]

\[ \tau_{\phi z} = 0 \quad \text{for} \quad r > a, \quad z = 0 \quad (3.10) \]

The solution of Equation 3.5, assigned this set of boundary conditions, must deliver the stresses \( \tau_{\phi z} \) and \( \tau_{\phi r} \) in the contact region or, equivalently, the torque \( M_v \) applied to the vibrating source. The interaction between the vibrator and the half-space medium is usually expressed in terms of the torsional mechanical impedance \( Z_T \), defined in Equation 3.11:

\[ Z_T = \frac{M_v}{j\omega \theta_0} \cdot \frac{1}{\pi a^2} \quad (3.11) \]

### 3.2.1 Analytical solution of the half-space problem

The first case analyzed is the classic half-space problem depicted in Figure 3.1. The solution can be found through integral Hankel transforms, reducing the problem to a set of dual Fredholm integral equations \([17, 50, 83]\). This boundary value problem for the Equation 3.5 with mixed (kinematic and kinetic) boundary conditions can be solved using a method described by Dorn \([17]\). The problem is conveniently treated using a dimensionless approach (the subscript 0 indicates dimensionless quantities), dividing all distances by the radius \( a \) of the contact area. Equation 3.5 is rewritten in the following form:

\[ \frac{\partial^2 u_0}{\partial z_0^2} + \frac{\partial}{\partial r_0} \left[ \frac{1}{r_0} \frac{\partial (ru_0)}{\partial r_0} \right] + k_0^2 u_0 = 0 \quad k_0 = \frac{\omega c_{SH}}{a} \quad (3.12) \]

where \( k_0 \) is the dimensionless wave number. This number characterizes the wave propagation pattern in the half-space and can be rewritten as in Equation 3.13:

\[ k_0 = \frac{\omega}{\sqrt{G^*/\rho}} R = \left( \frac{\omega}{\sqrt{|G^*/\rho|}} \right) e^{-j\frac{\delta}{2}} = |k_0|e^{-j\frac{\delta}{2}} \quad (3.13) \]

The relation between the shear stress and the displacement gradient in the contact area,

\[ \tau_{\phi z}(r, 0) = G^* \frac{\partial u(r, 0)}{\partial z} \quad (3.14) \]

can be rewritten by defining a dimensionless stress \( \tau_{0\phi z} = \tau_{\phi z}/G^* \) as follows:

\[ \tau_{0\phi z}(r_0, 0) = \frac{\partial u_0 (r_0, 0)}{\partial z_0} \quad (3.15) \]
3. Modeling the Interaction between Sensor and Material

The solution of the Bessel-type differential Equation 3.12 can be obtained using the Hankel transform [1], defined in Equation 3.16 as:

\[ \bar{g}(\xi, z) = H_1[g(r, z); \xi] = \int_0^\infty g(r, z) r J_1(\xi r) dr \]  

(3.16)

where \( J_1 \) is the Bessel function of first kind of the new Hankel-transformed variable \( \xi \) that substitutes \( r \) (functions with the bar symbol \( \bar{g} \) are defined in the Hankel space). Applying the \( H_1 \) transform to Equation 3.12, the elastodynamic wave equation can be simplified as:

\[ \bar{u}_0(\xi_0, z_0) = H_1[u_0(r_0, z_0); \xi_0] \]  

(3.17)

\[ \frac{d^2 \bar{u}_0}{dz_0^2} + (k_0^2 - \xi_0^2) \bar{u}_0 = 0 \]  

(3.18)

The solution of Equation 3.18, for a wave travelling toward the half-space interior, can be expressed as:

\[ \bar{u}_0(\xi_0, z_0) = A_0(\xi_0) e^{-\beta_0 z_0} \]  

(3.19)

where \( A_0(\xi_0) \) must be determined from the boundary conditions, and the term \( \beta_0 \) contains the information on the material properties. The boundary conditions in Equation 3.6 and 3.7 can be now expressed also using the Hankel transform and dimensionless quantities:

\[ u_0(r_0, 0) = H_1[\bar{u}(\xi_0, 0); r_0] = \theta_0 r_0 \]  

(0 \ < \ r_0 \leq 1)  

(3.20)

\[ \tau_{0zz}(r_0, 0) = H_1 \left[ \frac{d\bar{u}(\xi_0, 0)}{dz_0}; r_0 \right] = 0 \]  

(0 \ < \ r_0 \leq 1)  

(3.21)

Substituting the general solution of Equation 3.19 in Equations 3.20 and 3.21, the following pair of dual integral equations describes the problem:

\[ H_1[A_0(\xi_0); r_0] = \theta_0 r_0 \]  

(0 \ < \ r_0 \leq 1)  

(3.22)

\[ H_1[-\beta_0 A_0(\xi_0); r_0] = 0 \]  

(3.23)

The solution \( A_0(\xi_0) \) of the integral equations 3.22 and 3.23 can be found, as proposed by Gladwell [24], introducing a new function \( \Psi_0(x_0) \), using the Fourier sine transform \( \mathcal{F}_s \):

\[ \Psi_0(x_0) = \begin{cases} \mathcal{F}_s[\beta_0 A_0(\xi_0), x_0] & x_0 \leq 1 \\ 0 & x_0 > 1 \end{cases} \]  

(3.24)

\[ \mathcal{F}_s[g(t); x] = \sqrt{\frac{2}{\pi}} \int_0^\infty g(t) \sin(xt) dt \]  

(3.25)
Finally, the problem of a torsional vibrating source on a viscoelastic half-space, for 
the case of a linear displacement field in the contact area, is reduced to the following 
set of Fredholm integral equations of the second kind [24]:

\[
\sqrt{\frac{2}{\pi}} \theta_0 x_0 = \Psi_0 (x_0) + \int_0^1 \Psi_0 (x_0) M_0 (x_0, y_0) dy_0 
\] (3.26)

\[
M_0 (x_0, y_0) = \frac{2}{\pi} \int_0^\infty \left[ \frac{\xi_0}{\sqrt{\xi_0^2 - k_0^2}} - 1 \right] \sin(\xi_0 y_0) \sin(\xi_0 x_0) d\xi_0 
\] (3.27)

Equation 3.26 and 3.27 can be solved representing the unknown function \( \Psi_0 (x_0) \) 
as a finite series of normalized Legendre polynomials [17]. Once \( \Psi_0 (x_0) \) has been 
determined, \( A_0 (\xi_0) \) is found using inverse Fourier sine transform.

From the Hankel-transformed displacement of Equation 3.19, the displacement 
in the entire half-space is obtained by integration:

\[
u_0 (r_0, z_0) = H_1 [\bar{\nu}_0 (\xi_0, z_0); r_0] = \int_0^\infty A_0 (\xi_0) \xi_0 e^{-\sqrt{\xi_0^2 - k_0^2} z_0} J_1 (\xi_0 r_0) d\xi_0 
\] (3.28)

The dimensionless stress in the contact area can be determined from \( \Psi_0 (x_0) \):

\[
\tau_{\varphi z} (r_0, 0) = -H_1 [\mathbf{F}_s [\Psi_0 (x_0); \xi_0]; r_0] 
\] (3.29)

and the viscoelastic torque \( M_v \) applied to the vibrating source, or the torsional 
mechanical impedance \( Z_T \), are determined integrating the stress \( \tau_{\varphi z} \) in the contact 
area:

\[
M_v = 2\pi G^* \int_0^a r^2 \tau_{\varphi z} (r, 0) dr 
\] (3.30)

or equivalently:

\[
M_v = 2\pi a^3 G^* \int_0^1 r_0^2 \tau_{0\varphi z} (r_0, 0) dr_0 
\] (3.31)

**Comparison with the literature**

The torsional mechanical impedance of a medium can be determined for given values 
of the material parameters \( \rho, G^* \) and excitation frequency \( f \). Calculations were 
performed using *Matlab* (The MathWorks, USA), creating a function called TORIMP 
that provides torsional impedance, contact stress and displacement field. The 
calculations inside TORIMP involve the execution of several numerical integrals, as 
the Hankel- and sine-transform of Equation 3.16 and 3.25 on oscillating functions of 
complex value (due to the nature of the viscoelastic medium described by \( G^* \)). Due 
to the computational efforts required to solve Equations 3.26 and 3.27, especially 
at high material loss factors and high values of \( |k_0| \) (see [17] for more details), the 
function TORIMP is limited to an upper value of the dimensionless wave number
3. Modeling the Interaction between Sensor and Material

$|k_0| \approx 30$. This upper limit (three times higher than the one provided by Dorn in his work [17]) is appropriate for the range of frequencies and materials considered in this thesis. Within this limit, the solution of the elastodynamic problem can be found for any value of the loss factor $\eta = \tan \delta$ in the viscoelastic medium, from the nearly elastic solid $\eta \to 0$ to the perfect viscous fluid $\eta \to \infty$.

The values returned by the calculations in Matlab were compared for validation purposes to those tabulated by Dorn [17]. The comparison was made on the dimensionless torsional impedance $Z_{T0}$ defined in Equation 3.32:

$$Z_{T0} = \frac{Z_T}{\rho a^2 c_{SH}} \frac{3\pi}{16} = |Z_{T0}| e^{j\phi_T} \tag{3.32}$$

where the magnitude $|Z_{T0}|$ and the phase angle $\phi_T$ of this complex quantity have been defined. It is also convenient to report the results in terms of two coefficients, $R$ and $I$, often used in the literature [4, 17, 50, 72], the damping and stiffness coefficients, respectively, defined in Equation 3.33 as:

$$Z_{T0} = R - j \frac{I}{|k_0|} \tag{3.33}$$

These coefficients are usually given in tables as a function of the dimensionless wave number $|k_0|$, quantifying the effect of the viscoelastic medium on the vibrating source. The damping coefficient $R$ has always a positive value and represents the sum of all energy-absorbing effects in the medium (energy spent for SH-waves radiation and internal losses in the material). The stiffness coefficient $I$ includes elastic and inertial effects sensed by the vibrating source: a positive value of $I$ means that the medium behaves mainly as a “spring” increasing the stiffness properties of the mechanical system, while a negative value of $I$ means that the medium behaves as an inertia for the vibrating system.

Figure 3.3 shows a comparison between the values of the coefficients $R$ and $I$ tabulated by Dorn [17] and those evaluated using the function TORIMP. The comparison refers to a material loss factor of $\eta = 10^{-5}$ (nearly elastic solid, as considered in [17]) for $|k_0|$ varying from 0 to 10. In Figure 3.4, the dimensionless torsional impedance $Z_{T0}$ is plotted using the magnitude $|Z_{T0}|$ and the phase angle $\phi_T$ up to a value of $|k_0| = 30$. For values of $|k_0| \to \infty$, the vibrator essentially radiates plane waves in the near field [26], inducing a wave propagation mainly in the vertical direction $z$. In this case, the torsional impedance approaches the asymptotic value of plane wave impedance:

$$Z_T = \frac{1}{2} \sqrt{G^* \rho \cdot a^2} \tag{3.34}$$

Equation 3.34 can be used to calculate the torsional impedance above the upper limit $|k_0| \approx 30$ of TORIMP.
3.2. Problem statement: forced torsional oscillations on a viscoelastic half-space

![Graph](image1)

**Figure 3.3:** Comparison of the stiffness and damping coefficients $I$ and $R$, respectively. Solid lines represent the values extracted from Matlab using the function TORIMP, points represent the data tabulated by Dorn [17] for a material loss factor $\eta = 10^{-5}$ (nearly elastic solid).

![Graph](image2)

**Figure 3.4:** Dimensionless torsional impedance $Z_{T0}$: magnitude $|Z_{T0}|$ and the phase angle $\phi_T$ for a material loss factor $\eta = 10^{-5}$ (nearly elastic solid). Calculations up to a dimensionless wave number $|k_0| = 30$. 

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Figure 3.5: On the left hand side: calculated shear stresses at \( z = 0 \) for a medium with \( |k_0| = 0.5, 4 \) and 20, respectively, and \( \delta = 0.1 \). On the right hand side: corresponding radiation patterns of \( u_\varphi \). Calculations executed for \( a = 2.55 \) mm (TRD case), \( \rho = 1000 \) kg/m\(^3\), frequency of tests \( f = 1300 \) Hz, amplitude at the tip of the resonator \( a \cdot \theta_0 = 0.001 \) mm.
3.2. Problem statement: forced torsional oscillations on a viscoelastic half-space

Integration of the contact stresses and radiation pattern

The function TORIMP returns also the distribution of the contact stresses and the displacement field in the whole half-space, after solving Equations 3.28 and 3.29. On the left hand side of Figure 3.5, the components $\tau_{\varphi z}$ and $\tau_{\varphi r}$ of the stress vector at the tissue surface ($z = 0$) for a calculation with $|k_0| = 0.5, 4$ and $20$ are shown. On the right hand side of Figure 3.5, the corresponding radiation patterns are reported, with colors indicating the amplitude of the azimuthal displacement. The analytical solution leads to a stress singularity in correspondence to the external limit of the contact region $r \rightarrow a$. Sliding in the contact region must be reduced as much as possible in order to reproduce in the experiments the boundary conditions assumed for the analytical model, but cannot be completely avoided in correspondence to the borders of the contact region. The influence of sliding on the results of the test, depending on the size of the sliding area, will be quantified in Section 3.4.

The radiation pattern depends on the complex shear velocity $c_{SH}$, the angular frequency of excitation $\omega$ and the radius of the contact area $a$, which define the dimensionless wave number $k_0$ reported in Equation 3.13. This number characterizes the type of wave propagation pattern in a medium. For stiff materials at low frequencies, wave propagation in mainly radial direction is observed (Figure 3.5a, where $|k_0| = 0.5$). For soft materials and relatively high frequencies, $|k_0|$ assumes values higher than 3, leading to waves propagating mainly in $z$-direction, toward the half-space interior (Figure 3.5b, with $|k_0| = 4$, and Figure 3.5c, with $|k_0| = 20$).

The shear wavelength $\lambda_{SH}$ in the half-space is related to the dimensionless wave number by the relation:

$$\lambda_{SH} = \frac{2\pi}{Re(k_0)} a$$  \hspace{1cm} (3.35)

$$\left(\frac{a}{\lambda_{SH}}\right) = \frac{Re(k_0)}{2\pi}$$  \hspace{1cm} (3.36)

where $Re(k_0)$ is the real part of the complex wave number $k_0$. The ratio $\left(\frac{a}{\lambda_{SH}}\right)$ identifies the number of wavelengths contained in the radius of the vibrating source. For a nearly elastic case, $k_0 = 2\pi$ corresponds to a wavelength $\lambda_{SH} = a$. In the following sections, the torsional impedance is always studied as a function of $k_0$: Equation 3.36 can be used to interpret the results in terms of the wavelength $\lambda_{SH}$.

The attenuation of the SH waves in the half-space is determined by the phase angle $\delta$ of the complex shear modulus. The plots in Figure 3.5 correspond to $\delta = 0.1$. The material analyzed in this thesis show typically stronger viscous components, as in the case of the soft biological tissues and the synthetic materials considered in the following chapters (values of $\delta = 0.2 - 1.25$ corresponding to a material loss factor $\eta = \tan \delta = 0.2 - 3$). The amplitude of the displacement induced by the vibrating sensor decreases by one order of magnitude outside a region of 3 to 4 times the
3. Modeling the Interaction between Sensor and Material

oscillator diameter. As a consequence, the half-space assumption used to evaluate
the torsional impedance can also be used for samples of finite dimensions in which
the distance between radiating source and sample boundaries is in the range of 3-4
a.

3.2.2 Extension of the solution to a layered half-space

The analytical procedure described for a homogenous half-space can be extended
to a layered half-space, consisting of $N$ isotropic, linear viscoelastic layers, each
characterized by the thickness $q_m$ and material properties $G_m^*$ and $\rho_m$ with $m = 1...N$.

The general solution of the elastodynamic problem in the Hankel space (similarly
to what was presented in Equation 3.19 for a homogenous half-space), has now to
be found for each layer $m^{th}$ in the general form:

$$
\bar{u}_{0m}(\xi_0, z_{0m}) = A_{0m}(\xi_0)e^{-\beta_{0m} z_{0m}} + B_{0m}(\xi_0)e^{\beta_{0m} z_{0m}}
$$

(3.37)

$$
\beta_{0m} = \sqrt{\frac{\xi_0^2}{c_{SHm}^2} - k_{0m}^2} \quad k_{0m} = \frac{\omega}{c_{SHm}} a \quad c_{SHm}^2 = \frac{G_m^*}{\rho_m}
$$

(3.38)

where both coefficients, $A_{0m}$ and $B_{0m}$, are functions of the transformed radial vari-
able $\xi_0$ and $z_{0m}$ is the the vertical coordinate in each layer ($z_{0m} = z_m/a$), as shown
in Figure 3.6. On the right hand side of Equation 3.37, the first term represents the

Figure 3.6: Half-space consisting of $N$ isotropic, linear viscoelastic layers char-
acterized by the thickness $q_m$ and material properties $G_m^*$ and $\rho_m$ with $m = 1...N$.
down-going wave, the second term the up-going wave. At each interface between two different layers in the medium, the displacement $u_\phi$ and the shear stress $\tau_{\phi z}$ must be continuous, leading to the following conditions:

$$ u_0m(r_0, q_0m) = u_{0m+1}(r_0, 0) \quad q_0m = \frac{q_m}{\alpha} \quad (3.39) $$

$$ G'_m \frac{\partial u_0m(r_0, q_0m)}{\partial z_0} = G'_{m+1} \frac{\partial u_{0m+1}(r_0, 0)}{\partial z_0} \quad (3.40) $$

Applying the Hankel transform to the Equations 3.39 and 3.40, $u_0m$ and $r_0$ are simply replaced by $\bar{u}_0m$ and $\xi_0$, respectively:

$$ \bar{u}_0m(\xi_0, q_0m) = \bar{u}_{0m+1}(\xi_0, 0) \quad (3.41) $$

$$ G'_m \frac{\partial \bar{u}_0m(\xi_0, q_0m)}{\partial z_0} = G'_{m+1} \frac{\partial \bar{u}_{0m+1}(\xi_0, 0)}{\partial z_0} \quad (3.42) $$

The general solution of the elastodynamic problem reported in Equation 3.37 must now be substituted into Equations 3.41 and 3.42. This leads to an expression for the ratio $\gamma_0m$ (reflection coefficient) between the coefficients of the up-going and down-going waves in the $m^{th}$ layer, defined as follows [17]:

$$ \gamma_0m = \frac{B_0m}{A_0m} = \frac{\alpha_0m + \kappa_0m\gamma_{0m+1}}{\kappa_0m + \alpha_0m\gamma_{0m+1}} \cdot \frac{1}{\epsilon^{2\beta_{0m}q_0m}} \quad (3.43) $$

where the following quantities have been defined:

$$ \alpha_0m = (1 - \epsilon_{0m+1}) \quad (3.44) $$

$$ \kappa_0m = (1 + \epsilon_{0m+1}) \quad (3.45) $$

$$ \epsilon_{0m+1} = \frac{G'_{m+1}}{G'_m} \cdot \frac{\beta_{0m+1}}{\beta_{0m}} \quad (3.46) $$

In a medium consisting of $N$ layers, where the lower $N^{th}$ layer is a half-space, $\gamma_{0N} = 0$ because there is no up-going wave in the lower half-space. Starting with $\gamma_{0N} = 0$, the coefficients $\gamma_{0m}$, $m = N-1, N-2, \ldots, 1$ may be calculated recursively using Equation 3.43. The coefficients $\gamma_{0m}$ can be determined with simple algebraic equations once the characteristics of the medium are given (see also Equation 3.38 for the value of $\beta_{0m}$). Depending on the characteristics of two adjacent layers, these coefficients identify the importance of the wave reflection phenomenon at each interface.

The solution of the elastodynamic problem proceeds in a way similar to the one outlined for the homogenous half-space case. The stress distribution beneath the vibrating source must be solved, considering the first layer ($m = 1$) to identify the boundary value problem. The dimensionless displacement and stress in the first
layer, in correspondence to the surface \((m = 1, z = 0)\) are defined in the Hankel space as:

\[
\begin{align*}
\bar{u}_{01}(\xi_0, 0) &= A_{01}(\xi_0) + B_{01}(\xi_0) = (1 + \gamma_{01}) A_{01}(\xi_0) \\
\frac{d\bar{u}_{01}(\xi_0, 0)}{dz_0} &= \beta_{01}(\gamma_{01} - 1) A_{01}(\xi_0)
\end{align*}
\]  

(3.47)  

(3.48)

The boundary conditions can be formulated as follows:

\[
\begin{align*}
H_1[(1 + \gamma_{01})A_{01}(\xi_0); r_0] &= \theta_0 r_0 \quad 0 < r_0 \leq 1 \\
H_1[\beta_{01}(\gamma_{01} - 1)A_{01}(\xi_0); r_0] &= 0 \quad r_0 > 1
\end{align*}
\]  

(3.49)  

(3.50)

These equations correspond to a linear displacement field in the contact area and a stress-free surface outside it. The function \(\Psi_0(x_0)\) proposed by Gladwell [24] in his solution is now defined again using the sine transform \(F_s\) as:

\[
\Psi_0(x_0) = \begin{cases} 
F_s[\beta_{01}(1 - \gamma_{01})A_{01}(\xi_0); x_0] & x_0 \leq 1 \\
0 & x_0 > 1
\end{cases}
\]  

(3.51)

The set of Fredholm integral equations of the second kind that has to be solved in the case of a layered medium is:

\[
\sqrt{2\pi} \theta_0 x_0 = \Psi_0(x_0) + \int_0^1 \Psi_0(x_0)M_0(x_0, y_0)dy_0
\]  

(3.52)

\[
M_0(x_0, y_0) = \frac{2}{\pi} \int_0^\infty \left[ \frac{(1 + \gamma_{01})}{(1 - \gamma_{01})} \frac{\xi_0}{\sqrt{\xi_0^2 - k_{01}^2}} - 1 \right] \sin(\xi_0 y_0) \sin(\xi_0 x_0) d\xi_0
\]  

(3.53)

The solution \(\Psi_0(x_0)\) of the problem can be found again approximating this function using Legendre polynomials [17], and the coefficients \(A_{01}\) and \(B_{01}\) can be determined. From the boundary conditions at each layer interface, reported in Equations 3.39 and 3.40, all the coefficients \(A_{0m}\) and \(B_{0m}\) can then be calculated recursively starting from the \(A_{01}\) and \(B_{01}\).

The dimensionless stress in the contact area can be determined again from \(\Psi_0(x_0)\) using Hankel- and sine-transform:

\[
\tau_{0zz}(r_0, 0) = -H_1[F_s[\Psi_0(x_0); \xi_0] ; r_0]
\]  

(3.54)

The torque and the torsional impedance applied by the vibrating source are obtained through integration of the stresses in the contact region, as it was described for the homogenous half-space in Equations 3.30 and 3.31. The displacement field in the \(m^{th}\) layer can be found through integration:

\[
u_{0m}(r_0, z_0) = H_1[\bar{u}_{0m}(\xi_0, z_0); r_0] = \int_0^\infty \left[ A_{0m}(\xi_0) e^{-\sqrt{\xi_0^2 - k_{0m}^2}z_0} + B_{0m}(\xi_0) e^{\sqrt{\xi_0^2 - k_{0m}^2}z_0} \right] \xi_0 J_1(\xi_0 r_0) d\xi_0
\]  

(3.55)
3.2. Problem statement: forced torsional oscillations on a viscoelastic half-space

The presence of several layers in the half-space results into a complicated solution that has to consider the multiple reflections in the material due to the inhomogeneity of its structure. An inverse material characterization through dynamic torsion tests is impossible due to the many parameters that have to be estimated in a multi-layer model: this could be possible only using a spectroscopic measurement technique, i.e. executing measurements at several testing frequencies. Two simple cases are now presented to show the effect of a layered medium on the impedance function and on the possible application of the dynamic torsion test.

Solution for a unique finite layer

For the simple case of a unique finite layer \( (m = 1) \) of thickness \( q_{01} = 1 \), it is \( \epsilon_{0m+1} = \epsilon_{02} = 0 \), and therefore:

\[
\gamma_{01} = \frac{B_{01}}{A_{01}} = \frac{1}{e^{2\beta_{01}q_{01}}} \quad \beta_{01} = \sqrt{\varepsilon_0^2 - k_{01}^2}
\]

(3.56)

The solution of the elastodynamic problem is indeed a stationary solution, due to the finite dimensions of the layer thickness. Figure 3.7 shows the solution in terms of the dimensionless impedance \( Z_{T0} \), considering a nearly elastic layer (loss factor \( \eta_1 = 10^{-5} \)). The impedance curve presents several peaks, each representing a resonance of the layer. The resonance phenomenon takes place due to the reflection of plane waves in correspondence to the layer interface. At resonance, the shear wavelength \( \lambda_{SH} \) and the thickness \( q_1 \) must be linked by the following relation:

\[
n \cdot \lambda_{SH} = 2q_1
\]

(3.57)

with \( n = 1, 2, \ldots \infty \). With simple algebraic equations [17], it can be noticed that:

\[
\left( \frac{\lambda_{SH}}{a} \right) = \frac{2q_{01}}{n}
\]

(3.58)

\[
\left( \frac{\lambda_{SH}}{a} \right) = \frac{1}{n} \cdot \frac{2\pi}{Re(k_0)}
\]

(3.59)

\[
Re(k_0) = n \frac{\pi}{q_{01}}
\]

(3.60)

Equation 3.60 specifies the distance between two resonance peaks in Figure 3.7 (for nearly elastic layer is \( Re(k_0) \approx |k_0| \)). This distance decreases as the layer thickness \( q_{01} \) increases. For a wavelength \( \lambda_{SH} > 2q_1 \) (corresponding to \( Re(k_0) < \pi/q_{01} \)) no resonance can be observed. For the range of materials, frequencies and layer thickness of the material samples analyzed in this thesis (Sections 4.4.4 and 5.4), no resonance phenomenon was observed.
3. Modeling the Interaction between Sensor and Material

Figure 3.7: Dimensionless torsional impedance $Z_{T0}$ of a single layer of thickness $q_{01} = 1$ ($\eta_1 = 10^{-5}$, nearly elastic case to emphasize resonance peaks). The homogeneous half-space curve is reported for comparison.

Figure 3.8: Dimensionless torsional impedance $Z_{T0}$ of a (1) layer of thickness $q_{01} = 1$ on (2) half-space ($G_1 = 2G_2$, $\rho_1 = \rho_2$ and $\eta_1 = \eta_2 = 10^{-5}$). The homogeneous half-space curve is reported for comparison.
3.3. Finite element solution

Solution for a single layer over half-space

Considering now a layer of thickness \( q_{01} = 1 \) placed over a half-space, the results of the torsional impedance calculations are reported in Figure 3.8. The shear modulus of the top layer was chosen the double of the half-space value, with the same density \( \left( G_1^* = 2G_2^*, \rho_1 = \rho_2 \text{ and } \eta_1 = \eta_2 = 10^{-5}, \text{ nearly elastic case} \right) \). For comparison, the torsional impedance of the half-space alone is reported in Figure 3.8. It is evident that the presence of a layer will result into sinusoidal oscillations of the impedance around the half-space solution, representing the resonances of the top layer. The distance between the resonances is again determined by the thickness of the top layer, increasing with decreasing thickness.

3.2.3 Partially embedded vibrator

A similar procedure, based on the reduction of the problem to a set of Fredholm integral equations, can be used for the case depicted in Figure 3.2, where the torsional vibrating source is partially embedded into a medium for a height \( h \). The solution was first derived by Luco for the static case [50], then extended to the dynamics by Apsel and Luco [4], Rajapakse and Shah [72], Mita and Luco [57], with analytical and semi-analytical approaches. In the present work, no analytical solution was derived for this interaction case, but a more general finite element solution that is also suitable for the analysis of complicated contact geometries and consider the deformation of the torsional vibrating sensors. The finite element approach is presented in the following section, using the analytical solutions as a reference for validation purposes.

3.3 Finite element solution

The finite element method can be employed in studying the problem of forced torsional vibrations in a viscoelastic medium. An approach based on finite elements was developed using a commercial code, Abaqus (Hibbitt, Karlsson and Sorenson Inc., Pawtucket, USA). In this approach, the torsional mechanical impedance of a viscoelastic medium is determined as a function of its material properties and excitation frequency of the vibrating source. The proposed FE approach presents a series of advantages, with respect to the analytical solutions [4, 17, 50], in treating the problem of forced torsional vibrations in case of:

(i) particular sensor geometries and contact conditions between vibrator and medium, such as: non-cylindrical vibrators, curved surface of the samples, adherence only in a specified region of the contact area,

(ii) testing specimens with finite dimensions, where the half-space assumption usually required by analytical solutions is not valid,

(iii) inhomogeneous material samples, typically multi-layered materials,
3. Modeling the Interaction between Sensor and Material

(iv) deformability of the sensors in contact with the medium, which also has to be considered in case the material stiffness is high \((|G^*| > 10^7)\),

(v) nonlinear material behavior, when the vibration amplitudes exceed the linear limits or the material characteristics require a nonlinear constitutive model, or when the pre-deformation of the sample has to be considered.

3.3.1 Description of the FE approach with Abaqus

The problem described in Figure 3.1 and 3.2 was solved considering only a finite region of the half-space, discretizing it in finite elements. The commercial finite element code *Abaqus* was chosen for this analysis in order to exploit some useful built-in features, which can be of particular interest in handling viscoelasticity, nonlinearities and large strain deformations.

The problem of forced torsional oscillations was solved under the assumption of axis-symmetry. The use of two-dimensional axis-symmetric elements in *Abaqus*, that would have considerably simplified the solution, was not allowed due to the impossibility to consider this particular dynamic case, where the direction of the vibration is orthogonal to the axis-symmetric plane (only a static analysis is allowed with so-called TWIST elements: the inertia out of the plane is not considered). An alternative to the use of a commercial program was the formulation of a self-made FE code, based on a semi-analytical formulation of plane axis-symmetric elements [8]. This alternative was not considered, preferring the features already offered by a well established FE code as *Abaqus*, although the problem had to be modeled with 3D elements (numerically more expensive). This choice allowed an easier implementation of different material laws and the reliability offered by the *Abaqus* solver.

With reference to Figure 3.9, the problem of forced torsional oscillations was studied using three-dimensional elements (second order brick element type C3D20H in *Abaqus*). The full three-dimensional problem was simplified by introducing a cyclic-symmetry boundary condition to an angular section (as depicted in Figure 3.9), with a clear advantage in terms of nodes and elements number. Even though the problem is formulated in 3D, the cyclic-symmetry boundary conditions reduces it to an axis-symmetric problem. This assumption allows a reduction of the number of degrees of freedom and a faster solution of the model. The models considered in this work consisted of a maximum number of nodes of about 100000 and could perform a single torsional impedance calculation within 20 minutes on a 1.5 GHz computer with 1 GB RAM.

The axis-symmetric assumption does not allow to consider models whose properties are not uniformly distributed along the \(\varphi\) coordinate, such as anisotropy of the sample or inhomogeneities that are not constant in the azimuthal direction (e.g. the presence of an inclusion of different stiffness properties in the model, such as a spherical cavity). Nonetheless, the finite element approach proposed here could be also used to solve full, complex 3D problems (without cyclic-symmetry boundary
3.3. Finite element solution

Angular Section
Mesh

\[ \theta = \theta_0 e^{j\omega t} \]

Figure 3.9: Axis-symmetric problem of forced torsional vibrations: the problem is solved on an angular section with cyclic-symmetric boundary conditions.

condition, extending the angular section to a full 360 degrees geometry): this would require more computational efforts, since the level of discretization required for a small angular section must be repeated for the entire 360 degrees-model.

In the proposed finite element approach, the material law can be prescribed to be linear viscoelastic, specifying values of the storage and loss shear moduli \( G_1 \) and \( G_2 \) that can be frequency dependent. The use of an hyperelastic material law for modeling the medium is also allowed and can be useful to study the effect of pre-deformation on the results. The mesh refinement must be changed according to the characteristics of the material analyzed, in order to correctly model the SH-wave propagation in the medium and the stress concentration that always occurs at the external boundary of the vibrator. The wavelength \( \lambda_{SH} \) in the medium is related to the elastic properties of the material and to the frequency of excitation. Hence, a high level of discretization is required for soft materials excited at high frequencies: in case of tests of soft biological materials executed at high frequency, wavelengths in the order of magnitude of millimeters are expected (\( \lambda_{SH} = 0.2 - 5 \text{ mm for } f = 1-12 \text{ kHz} \)).

In order to verify the convergence of the results, calculations may be run with different mesh refinements. To facilitate the convergence check, the mesh of the model was automatically generated using a Matlab (The MathWorks, USA) routine instead of Abaqus. The possibility to build standard ”angular section” elements (see
Figure 3.10: On the left, assembly of different standard elements in the mesh to define the complete interaction model (HFTR sensor for field tests, parts 1 to 6, and viscoelastic material, 7 and 8). On the right, amplitude of the normalized displacement field $|u_\varphi|$ ($|u_\varphi| = 1$ at the resonator’s tip) of the torsional vibrating system during a test.

Figure 3.10) and to assemble them into a complete model (each section characterized by different material properties and geometry) makes the Matlab routine a flexible tool to be used in the analysis of torsional vibrating sensors.

The modular architecture of the mesh generation algorithm allows the analysis of complex sensors geometries, as in Figure 3.10, on the left, where the full model of the HFTR sensor for field tests (described in the Section 2.3.2) is shown. It consists of different parts, whose characteristics can be specified separately (material properties, interaction with other components, etc.): different colors indicate the sensor (1-6, made by different sections welded one to another) and the material in contact (7-8). In Figure 3.10, on the right, the displacement field $|u_\varphi|$ is calculated, with colors indicating the normalized amplitude of the displacement ($|u_\varphi| = 1$ at the tip of the vibrator). The different components 1-8 of the model can be bonded one to another (in case of perfect adherence), or a coefficient of friction can be defined.
between two contact surfaces. However, perfect adherence between vibrator and viscoelastic medium was assumed in the following analyses, as it is also assumed by the analytical solutions used afterwards as a reference.

Once the mesh has been generated and the boundary conditions defined, a data file generated by Matlab is solved with Abaqus. The vibrator can be considered as an elastic or rigid body, excited with a torque \( M_e \) at a given frequency \( f \) that drives the torsional vibrations. A steady-state dynamic analysis of the model is run to find the response of the mechanical system to harmonic excitation, with a frequency spectrum defined by the user (bandwidth and step must be specified). The solution is given in terms of displacement and stress fields in the whole model. The torsional impedance \( Z_T \) and the radiation pattern can be determined as a function of the input parameters \( Z_T(G^*, \rho, f, \text{contact geometry}) \).

### 3.3.2 Convergence analysis and validation of the FE approach

Some numerical challenges exist in the FE approach presented above, which is a complex harmonic analysis, involving calculations of viscoelastic materials in a wide frequency spectrum. Due to the shear-waves wavelength in the viscoelastic medium, which can be very small (for soft tissues tested at high frequencies it can be \( \lambda_{SH} \approx 0.2 \))

![Figure 3.11: Example of a convergence analysis for the FE approach proposed. Comparison between the value of \(|Z_{T0}|\) obtained by the FE approach to the reference value of \(|Z^0_{T0}|\) evaluated analytically by TORIMP for the case of rod-shaped vibrator over half-space, with \(|k_0|=4\) and \(\tan \delta = 0.3\). Two different meshes considered: uniform (on the right) and biased (on the left). \(N_i\) lines indicate solutions obtained with the same number of elements.](image-url)
mm), and to the stress concentration in correspondence to the external radius of the vibrator, a high level of discretization of the medium is required to correctly model this interaction. A convergence analysis of the results delivered by the FE solution is necessary to verify its reliability, running several FE calculations with different mesh refinements until the solution stabilizes within a certain tolerance.

An example of a convergence analysis is presented in Figure 3.11, considering the standard case of a rod-shaped vibrator placed on the top of a viscoelastic half-space. In the example presented here, \(|k_0|=4\) and the damping in the half space is introduced with the material loss factor \(\eta = \tan \delta = 0.3\). The half space can be discretized in finite elements using a uniform mesh, in which the element size is constant in the whole model, as shown in Figure 3.11, on the right. As alternative, a biased mesh can be used, in which the element size is smaller in correspondence to the region characterized by the stress concentration (in Figure 3.11 on the left, arrows indicate the direction of higher element density). In both cases, the maximum size of the elements (i.e. the largest side of the brick elements C3D20H) is identified by the parameter \(\chi_{\text{max}}\).

The results of calculations of the dimensionless torsional impedance \(|Z_{T0}|\) obtained with the FE approach are compared in Figure 3.11 to those obtained by the analytical solution with TORIMP \(|Z^0_{T0}|\) that is chosen as a reference for this standard case. The convergence analysis is reported in Figure 3.11 as a function of the ratio \(\lambda_{SH}/\chi_{\text{max}}\), by comparing the element size to the shear wavelength. Obviously, at high values of \(\lambda_{SH}/\chi_{\text{max}}\) (smaller element size), the FE solution approaches the analytical solution chosen as a reference. From Figure 3.11, it is evident that the convergence to the reference solution is faster with a biased mesh, that can better model the region where the stress concentration occurs. By considering FE calculations with the same number of elements \(N_i\) (indicated in Figure 3.11 by connecting lines with equal \(N_i\)), a better solution is obtained through a higher discretization of the critical regions with a biased mesh. In this thesis, the interaction between vibrator and viscoelastic medium is solved with FE using biased meshes instead of uniform.

The element size in the FE model \(\chi_{\text{max}}\) must be chosen to minimize the error committed on the torsional impedance evaluation below a specified tolerance. The numerical code must be able to evaluate the torsional mechanical impedance with an error of less than 1%. This limitation is required for the accuracy of the inverse material characterization from the experiments (the error propagation on the estimated shear modulus \(|G^*|\) is quadratic, as it will be later discussed). The maximum element size \(\chi_{\text{max}}\) was always chosen to guarantee this level of precision. The curves reported in the example of Figure 3.11 vary depending on the value of \(|k_0|\) and \(\tan \delta\) of the material interacting with the vibrator. A general criterion that links the element size \(\chi_{\text{max}}\) to the precision of the FE calculation could not be formulated. Therefore, in those cases where no analytical solution is available for comparison, a convergence check on the impedance provided by the numerical code must always be carried out, until the variation \(\Delta |Z_{T0}|\) due to a mesh refinement stabilizes within a specified tolerance.
3.3. Finite element solution

Figure 3.12: Magnitude $|Z_{T0}|$ of the dimensionless torsional impedance for the rod-shaped vibrator over half-space, comparison of the FE solution (solid lines) with the analytical solution discussed in Section 3.2.1 (markers), for three different values of loss factor $\eta = \tan \delta$ in the material.

Figure 3.13: Phase $\phi_T$ of the dimensionless torsional impedance for the rod-shaped vibrator over half-space, comparison of the FE solution (solid lines) with the analytical solution discussed in Section 3.2.1 (markers), for three different values of loss factor $\eta = \tan \delta$ in the material.
3. Modeling the Interaction between Sensor and Material

![Graph](image)

**Figure 3.14:** Comparison of the damping and stiffness coefficients $R$ and $I$, respectively, obtained from the analytical solution by Apsel [4] and Rajapakse and Shah [72] (markers) and obtained from the FE model (solid lines) for two values of the embedment ratio $h/a$. Values of the analytical solution only available up to $k_0 = 2$ and for elastic material, $\tan \delta = 0$.

The reliability of the proposed FE approach was verified by comparing the results obtained with FE to those obtained by the analytical solutions discussed in Section 3.2.1 for different values of $|k_0|$ and $\tan \delta$. First, the problem of a rod-shaped vibrator placed on the top of a viscoelastic half-space, depicted in Figure 3.1, was considered. In Figure 3.12 and 3.13, the dimensionless torsional impedance values $Z_T$ are compared in function of $|k_0|$. Three different values of the material damping were considered by specifying the value of $\eta = \tan \delta$, the loss factor in the material. In Figure 3.12 and 3.13, markers represent the analytical solution discussed in Section 3.2.1, while solid lines represent the results of the FE calculation (solid lines well represent the continuity of the FE calculations due to the high resolution chosen in the step for $k_0$). The two solutions are coincident in the range considered, the difference being always less than 1% in magnitude at least up to $|k_0| = 10$.

Considering the case depicted in Figure 3.2, rod vibrating source partially embedded into a medium, the comparison is made with different values of the embedment ratio $h/a$ and $k_0$, using the data available in the literature in the works of Apsel [4] and Rajapakse and Shah [72], for elastic material ($\tan \delta = 0$). The values evaluated with the FE approach agree to a great extent with the analytical ones, as reported in Figure 3.14.
3.3.3 Capabilities of the FE model

In the previous section, the reliability of the FE approach proposed has been proofed with a comparison to analytical solutions for some standard cases. This approach gives now the possibility to study cases that can be difficulty or not at all handled with analytical solutions. Different configurations that can often occur during experiments with the dynamic torsion test are analyzed and discussed here.

Effect of a lateral contact

One of the major sources of uncertainty in a dynamic torsion test is represented by the influence of an unwanted lateral contact on the vibrating sensor. Most of the experiments presented in this work were executed by placing the circular area at the tip of the resonators in contact with a material sample. If a contact on the lateral sides of the resonators occurs due to partial penetration, this has important consequences on the outcomes of the test. In testing soft biological tissues, the deformability of the material can also lead a partial contact on the sides of the resonator that can influence the results of the experiment. Although the TRD does not penetrate the soft tissue samples, these can partially adhere to the sides in case the device is pushed against them or is not perpendicular to them (typically, lateral contacts in the order of 0.1 mm can be observed).

A contact with a viscoelastic medium on the lower and on the lateral surface of the vibrator can easily be modeled with the finite element approach described in the previous section. The quantity $h$ identifies the height of a uniformly distributed

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure3.15.png}
\caption{FE simulation of a rod-shaped vibrator partially embedded in a viscoelastic medium. Magnitude of the normalized displacement amplitude $\|u_\phi\|$ (|$u_\phi$| = 1) at the tip of the vibrator.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure3.16.png}
\caption{Magnitude of the dimensionless torsional impedance for different values of the embedment ratio $h/a$ divided by the reference value with $h/a = 0$. Curves represent results obtained for different values of $|k_0|$ for a material loss factor $\eta = 0.3$.}
\end{figure}
lateral contact and $h/a$ is defined as the embedment ratio. In Figure 3.15, the FE model used for this analysis is shown. Figure 3.16 shows the results of calculations executed at different values of the embedment ratio $h/a$, reporting the results for three different values of the dimensionless wave number $k_0$, for a constant material loss factor $\eta = 0.3$. The magnitude of the dimensionless torsional impedance is compared to the reference value of $|Z^0_T|$,

where $h = 0$ and the contact lays only on the lower surface of the vibrator (corresponding to the analytical half-space solution). It is evident how critical the influence of a lateral contact can be. Considering $|k_0| = 5$ (soft material, high frequency) and $h/a = 0.25$, the magnitude of the impedance is already doubled with respect to the reference case. The phase angle is not considerably influenced by the lateral contact and was not reported here. The lateral contact assumes a higher importance at high values of $|k_0|$ and, assuming the same testing frequency, is critical in testing soft materials.

An overestimation of the mechanical impedance $|Z_T|$ by a factor of 2 would lead indicatively to an overestimation of the shear modulus $|G^*|$ by a factor of 4. Even though a direct relation between $Z_T$ and $G^*$ cannot be obtained in closed form (the torsional impedance is in general evaluated through integrals), useful information can be extracted from Equation 3.34, where the asymptotic formula (valid for $k_0 \to \infty$) for the torsional impedance was reported. This relation can serve as a rule of thumb to determine the error committed on the estimation of $|G^*|$ that goes with $|Z_T|^2$. For this reason, the TRD developed for testing soft biological tissues must reduce as much as possible the influence of a contact on the lateral sides (or at least control it very precisely, but this is difficult to realize). For a radius of the contact area $a = 2.55$ mm, a lateral contact of 0.1 mm turns already into an overestimation of $|G^*|$ of about 40%.

**Effect of the curvature of the samples**

The viscoelastic medium in contact with the vibrating sensor was assumed to be a half-space with planar surface. In reality, especially in testing solids whose geometry is not well defined, the surface can have a typical curvature that could influence the results. This situation is represented in Figure 3.17, where the vibrator of radius $a$ is placed on the top of a material sample whose surface has a constant radius of curvature $R_v$.

Perfect adherence is assumed at the interface between vibrator and medium for a radius $r \leq a$. In Figure 3.18 the magnitude of the torsional impedance obtained from FE calculations is compared to the reference one (planar surface). The curvature of the surface does not have a significant impact on the impedance. As long as the materials are soft (values of $|k_0| > 3$ and wavelength $\lambda < a/2$), the region interested by the wave propagation is confined into a small region close to the vibrator and is not affected by the curvature. Higher influences are expected for stiffer materials, while an increase of the internal material damping minimizes this effect (the curves in Figure 3.18 were obtained with a loss factor in the material $\eta = \tan \delta = 0.1$). Calculations were executed for $R_v/a > 4$: for smaller radius of curvature the finite
3.3. Finite element solution

Figure 3.17: FE simulation of contact with an axis-symmetric medium with curved surface. Curvature of the surface indicated by the radius $R_v$.

Figure 3.18: Influence of a curved surface with radius $R_v$ on the magnitude of the torsional impedance for three values of $|k_0| = 0.5$, $2$, $5$ and $\eta = 0.1$.

Figure 3.19: FE simulation of contact with a sample of finite dimensions. Cylindrical sample of diameter $d$ and height $d$ constrained at the bottom in the typical configuration of a dynamic torsion test.

Figure 3.20: Magnitude of the torsional impedance for different sample sizes $d/2a$. Curves represent results obtained for different values of $|k_0| = \frac{\omega}{\sqrt{G/\rho}}$ and for a loss factor $\eta = 0.3$.

dimensions of the sample play an important role as it will be further discussed.

Effect of sample size

The effect of the samples size can be investigated with the FE approach in order to give useful indications on the minimum size of the samples prior to executing the
3. Modeling the Interaction between Sensor and Material

tests and later to check the results. In Figure 3.19, a cylindrical sample of diameter \(d\) and height \(d\) in contact with a vibrator of radius \(a\) is considered. As usual, perfect adherence is assumed at the interface between vibrator and medium in contact. The lateral surfaces of the sample are not constrained, while the lower surface is fixed to the ground. In the typical configuration observed during a dynamic torsion test, the sample is laid on a table and fixed to its lower surface, while the lateral surfaces are left free to move. The magnitude of the torsional impedance is compared again with the reference value of the half-space solution for different values of the ratio \(d/2a\) and for three values of \(k_0\), choosing a value of the internal damping by specifying the loss factor \(\eta = 0.3\).

The results of these calculations are reported in Figure 3.20. The influence of the sample size is obviously higher for low values of \(|k_0|\) and higher \(\lambda_{SH}\). The oscillations shown in the curves correspond to resonances of the samples. In correspondence to a resonance, the impedance varies strongly: this situation must be avoided by choosing larger samples. The internal loss factor \(\eta\) of the viscoelastic medium affects strongly the results by increasing the attenuation of the waves, reducing the minimum size required for the samples.

### 3.4 Discussion

In this chapter, the different possibilities to model the interaction between a torsional vibrating sensor and a viscoelastic medium have been presented. There are few aspects that have to be considered to apply these models to an inverse material characterization and to develop and design sensors based on the same working principle.

**Sliding in the contact area**

It is evident from the behavior of the stresses \(\tau_{\varphi z}\) (shown in Figure 3.5) that the singularity observed for \(r \to a\) can result into sliding at the external border of the contact area between the resonator and the medium analyzed. Sliding cannot be completely avoided, but the sliding radius \(r_s\) must be as close as possible to \(a\) in order to reduce the uncertainty of the measurements. Considering the problem of a rod-shaped vibrator placed on a half-space, the dimensionless torsional impedance \(Z_{T0}\) varies with the sliding radius \(r_s\) according to the behavior presented in Figure 3.21. A value of \(r_s/a = 0.97\) (sliding on the external 3% of the radius) could result into an error of 25% on the impedance evaluated and of 56% on the magnitude of the shear modulus estimated. This phenomenon is more evident at high values of \(|k_0|\), due to the sharpness of the stress distribution observed in these case in correspondence to the external radius (see Figure 3.5, left, for comparison of the stress distribution).

The disc with micro-openings bonded in the contact area of the TRD was designed to reduce this sliding phenomenon. Through these openings, vacuum clamping minimizes the size of the sliding area. These openings were designed in the
Figure 3.21: Effect of sliding in the contact area (sliding for \( r_s < r < a \)) on the magnitude of the dimensionless torsional mechanical impedance \( Z_T^0 \), considering three different cases with \( |k_0| = 0.5, 2, 5 \) with \( \eta = 0.3 \).

Optimization and parameter extraction

The analytical and finite element solutions allow to determine the storage and loss modulus of a material starting from the value of the torsional mechanical impedance \( Z_{T,meas} \) measured in experiments with the TRD or HFTR devices. In Figure 3.22 and 3.23, the relation between the material parameters of the medium analyzed, \( |G^*| \) and \( \eta = \frac{G_2}{G_1} \), and the torsional impedance magnitude \( |Z_T| \) and phase \( \phi_T \) are reported for the half-space case. These surfaces represent the results obtained with the function TORIMP (that provides an analytical solution) for a given frequency of the test (in this case \( f = 1300 \) Hz, which corresponds to the first operative frequency of the TRD). Similar results can be obtained for all the other contact configurations considered and operative frequencies.

The use of an analytical solution allows a fast determination (few seconds) of the relation between the material parameters and the torsional impedance \( Z_T \). Starting from the values of \( Z_{T,meas} \) measured with experiments, a simple optimization procedure is then executed, searching the values of \( G_{1,\text{opt}} \) and \( G_{2,\text{opt}} \) that minimize the function:

\[
\min ||Z_T(G_1, G_2, \rho, f) - Z_{T,meas}|| = G_{1,\text{opt}}, G_{2,\text{opt}}
\]  

(3.61)
3. Modeling the Interaction between Sensor and Material

**Figure 3.22:** Magnitude of the torsional impedance $|Z_T|$ in function of the material parameters of the viscoelastic medium analyzed (half-space assumption, $f = 1300$ Hz, $a=2.55$ mm).

**Figure 3.23:** Phase of the torsional impedance $\phi_T$ in function of the material parameters of the viscoelastic medium analyzed (half-space assumption, $f = 1300$ Hz, $a=2.55$ mm).
\[ Z_{T,\text{meas}} = Z_{T,\text{meas}}(\Delta f_{\text{res}}, df) \]  

In Equation 3.62, the quantities measured during a test (\( \Delta f_{\text{res}}, df \)) are linked to the torsional impedance according to the procedure described in Section 2.2.3 with an adequate mechanical model of the sensors.

The use of a half-space analytical solution represents, in most of the cases, the easiest way to extract the material parameters, if the dimensions of the probe and the contact configuration are consistent with this assumption. As an alternative, the FE solution provides a reliable method to treat cases where the analytical solution cannot be used, but requires more computational time. In this case, several FE calculations are run in order to build tabulated values of the impedance \( Z_T(G_1, G_2, \rho, f) \), which can be then fitted by interpolating functions and later used in the optimization procedure described by Equation 3.61.
3. Modeling the Interaction between Sensor and Material
Chapter 4

Characterization of Soft Biological Tissues

This chapter deals with the mechanical characterization of soft biological tissue with the torsional resonator device. The measurement technique is first validated with comparative measurements performed on silicone and rubber phantoms with wave propagation methods. A comparison with the results obtained by other quasi-static and dynamic methods on a silicone phantom, the so called Truth Cube 2, are also presented. Results obtained from experiments performed ex vivo on bovine organs such as liver, kidney and uterus, demonstrate the suitability of the dynamic torsion test for measuring the mechanical behavior in shear. Soft internal organs are treated as homogenous isotropic materials with linear viscoelastic behavior, and their complex shear modulus $G^*$ is obtained in the frequency range 1-12 kHz. The influence of the capsule, the thin membrane that covers these organs, on the outcomes of the measurement is discussed. A set of comparative measurements on soft internal organs with a quasi-static method, the Aspiration Device, is provided, showing the possibility to fully characterize soft tissues at different strain and load rates. Final discussion about repeatability of the measurements, error sources and critical factors that affect the TRD are presented here.

4.1 Introduction

The identification of the mechanical properties of soft biological tissues is essential to a number of medical applications, such as for diagnostic purposes, surgery planning and training of surgical procedures with virtual reality-based simulators [6, 27, 69, 86]. Mechanical modeling of these materials is a challenging task, due to their typical characteristics of exhibiting nonlinear, viscoelastic behavior and to the capabilities
of undergoing very large strains. In most of the cases, the result of a test executed on a soft tissues sample depends on the direction of loading, because of anisotropy, and on the location, because of the inhomogeneities within the same tissue and among different individuals. Furthermore, their mechanical properties are influenced by environmental factors such as hydration, humidity, temperature, blood perfusion [22, 38, 94].

Among all the methods proposed by researchers willing to characterize biological tissues, two different approaches can be identified: (i) quasi-static and (ii) dynamic testing. Quasi-static experiments typically provide information on the nonlinear viscoelastic response for large deformations at low deformation rates, which is useful for prediction of organ deformations under physiological loads or simulation of surgical procedures. A number of different procedures for quasi-static tissue testing have been recently developed. They are based on indentation, aspiration or shear testing [30, 37, 55, 60, 65, 100].

Dynamic testing at higher strain rates provides additional information on the constitutive behavior of the tissue, with applications in diagnostics and trauma research [84]. Methods for dynamically testing soft biological materials range from standard rheometers operating at 0.01 to 10 Hz [47, 59], to devices suitable for characterization up to 350 Hz [5]. Techniques suitable for in vivo measurements offer the great advantage of a characterization in the natural biochemical environment with blood supply and hormone stimuli, which influence the mechanical properties of the tissue in a significant way. In the work of [42] and [51], porcine tissues were tested ex vivo and in vivo from 50 to 300 Hz using magnetic resonance elastography. Rotary shear tests have been proposed for in vivo tests by [36] for the low frequency range (up to 20 Hz).

In the novel measurement method proposed in this work, the mechanical properties of soft tissues are derived from the material response in shear at high frequencies (1-10 kHz) and small strains (up to 0.2%). In the dynamic torsion test, the vibration amplitude is kept below 0.001 rad and limits the shear strains to $\gamma_{\text{max}} < 0.2\%$, for the materials and range of frequencies considered here. Within this range, linear viscoelasticity can be assumed to describe the tissue behavior in shear: in agreement with the results found by Liu et al. [47] and Nasseri et al. [59], at this strain amplitude the tissue response is assumed to be linear viscoelastic. Characterization of the rheological properties of a tissue, in terms of a frequency dependent complex shear modulus $G^*$, can be obtained from the dynamic torsion test only for homogeneous and isotropic tissues. In fact inhomogeneities (e.g. a layered tissue) or anisotropy (in the plane of the tissue surface and perpendicular to it) lead to a larger number of material and geometry parameters which cannot be uniquely determined from the two experimental data $\Delta f_{\text{res}}$ and $df$, as it was discussed in Chapter 3. According to these assumptions, the complex shear modulus of the samples analyzed can be derived from the experimental results using the analytical model of a torsional vibrating source on a semi infinite space [17, 73, 77, 83] described in Chapter 3.

In this Chapter, the capabilities of the torsional resonator device (TRD) developed to test soft biological tissues are shown. The measurement is fast and, due to
the small contact area, a local characterization of the mechanical properties can be achieved. A mapping procedure and reference tables enable very fast (almost "on-line") determination of the material parameters. This technique does not require specific sample preparation, can be applied to samples of virtually arbitrary shape and uses simple, portable and inexpensive components.

The experimental procedure of the TRD in testing soft biological tissues is presented in Section 4.2, providing a proof of the non-slip condition, assumed in the analytical model to extract the materials parameters, and validating the measurements with comparative measurements on soft synthetic materials, for which viscoelastic properties were also measured with wave propagation experiments. Tests on a silicone phantom, the so called Truth Cube 2, have been executed and compared to the results obtained by other quasi-static and dynamic methods. The results obtained showed a good agreement with those obtained by other methods, and could serve as a reference for validating similar devices for soft tissue testing.

Results of measurements on bovine soft organs ex vivo (liver, kidney, uterus) are reported in Section 4.4. The repeatability and sensitivity of the TRD in testing soft biological tissues are discussed. A frequency dependent characterization of the linear viscoelastic properties of soft internal organs is provided and compared to the results obtained with quasi-static aspiration experiments [60]. The influence of the capsule, a stiffer thin membrane that covers soft internal organs such as liver and kidney, on the parameters estimated with the TRD is analyzed. A discussion on the error sources and on the possible improvements of this measurement technique is provided in Section 4.5.

### 4.2 Experimental procedure with the TRD

The Torsional Resonator Device (TRD) was developed to test soft biological tissues, typically soft internal organs. A detailed description of the device design was presented in Section 2.3.1. The device was used in the laboratory in its benchtop configuration shown in Figure 2.5, controlling the vertical position with respect to the material sample analyzed and using a balance to control the vertical force applied to it in order to avoid pre-deformations and pre-stresses.

In the standard procedure for ex vivo tests, a portion of a tissue sample is excised from the whole organ, placed into a container (15 cm × 15 cm × 10 cm box) and laid on the balance. The TRD position is controlled manually by two travel stages that give the possibility to adjust the vertical and horizontal position of the device with respect to the material sample. With reference to Figure 4.1, (a) the tip of the resonator is first pushed against the tissue, (b) the internal pressure of the tube is set to $p = 0.3$ bar by opening a valve, obtaining the vacuum clamping, (c) the TRD is pulled up, to ensure that the contact with the tissue lays only on the circular area of radius $a = 2.55$ mm and not on the lateral surfaces of the resonator, (d) the vertical position is finally adjusted until no vertical force is registered by the balance, and a measurement of $df$ and $\Delta f_{res}$ is executed.
Vacuum clamping is used to ensure adherence between the TRD and the soft tissues considered in order to fulfill the boundary condition assumed in the models that describe the interaction with the viscoelastic medium. Measurements of the velocity field at the surface of a biological tissue sample were carried out in the vicinity of the tip of the resonator in order to verify the reliability of the non-slip assumption. Retro-reflecting spheres were distributed on the sample surface and the azimuthal velocity field \( v(r) = \dot{u}_\phi(r) \) determined by laser interferometry. Figure 4.2 shows that the velocity field predicted using the analytical model (with non-slip assumption) agrees to a great extent with the measured values, whereas significant deviations are found with respect to a calculation assuming free sliding for the region \( 0.9 < r/a < 1 \) and \( 0.95 < r/a < 1 \). These results support the validity of the non-slip assumption for the TRD sensor.

It has been noticed that the application of the underpressure for a 10 seconds interval is sufficient to ensure a good clamping between the tissue surface and the TRD (similar to the type presented in Figure 4.2), for the animal tissues analyzed (bovine liver, kidney, uterus). The soft tissue samples stick very well to the contact disc by closing all the micro-openings. The tissue is not damaged by this procedure, as proofed by the absence of bleeding and abrasions in the contact area. Even after releasing the pressure inside the tube to ambient values, the tissue sample remains in contact with the sensor: the TRD has to be pulled up for some millimeters in order to detach it from the tissue surface. This good clamping obtained in a short period of time has also important consequences for the quality of the test: applying an underpressure continuously for longer time would result into dehydration of the tissue and consequently into a rapid change in the mechanical properties measured.

On the contrary, the same procedure cannot be applied to synthetic materials (as the silicone and rubber samples also considered in this chapter). For these materials, the underpressure must be applied continuously over the entire duration of the test to prevent sliding, that suddenly occurs after step (c) if the pressure is released. However, the properties of the synthetic samples analyzed were not at all affected by the prolonged application of the underpressure, the values of \( df \) and \( \Delta f_{res} \) being stable in time.
4.2. Experimental procedure with the TRD

Figure 4.2: Velocity field measured on the surface of a bovine liver sample with laser interferometry (experiment shown above right). Measurements are compared with the prediction of the analytical half-space model assuming non-slip condition, sliding for the region $0.9 < r/a < 1$ and sliding for $0.95 < r/a < 1$. Error bars in the plot represent the uncertainty of the measurement points.

The interaction between the TRD and the material sample was modeled using a half-space assumption, with the analytical solution described in Section 3.2.1. The dimensions of the portion of excised tissues were always big enough to be consistent with this assumption. From Figure 4.2 it is also evident that the SH-waves induced by the TRD are strongly damped within a region of radius 15 mm: the tissue samples considered had always a larger size and no stationary effect due to waves reflections could be observed.

By solving the analytical problem, the torsional impedance of the soft tissue sensed by the resonator can be expressed as a function of the material parameters. We assume that the density $\rho$ of the tissue is known ($\rho = 1030 \text{ kg/m}^3$, as commonly assumed in the literature [22]). Thus, the changes in the dynamic behavior of the resonator (the damping characteristic $df$ and the resonance frequency shift $\Delta f_{res}$) can be linked to the viscoelastic properties of the tissue by using the mechanical model of the sensor (reported in Appendix A.1) and the analytical half-space solution. The relationship between the material parameters $G_1$, $G_2$ and the measured quantities $df$ and $\Delta f_{res}$ can be directly determined with a mapping procedure, prior to executing the experiments.

The expected values of $df$ and $\Delta f_{res}$ related to specific values of $G_1$ and $G_2$ can be tabulated and later used during the experiments to quantify the material parameters almost on-line. In Figure 4.3, the relationship between the material parameters and the readings of the measurement are reported in form of a 3D plot.
Figure 4.3: Resonance frequency shift $\Delta f_{\text{res}}$ (on the left) and damping characteristic $df$ (on the right) in function of $G^*$ for the first resonance frequency ($f^{(I)} = 1300$ Hz). Values are reported for values of $|G^*| = 10^3 - 10^6$ Pa, from those expected for soft biological tissues to those typical of rubbers and silicones.

Figure 4.4: Resonance frequency shift $\Delta f_{\text{res}}$ (on the left) and damping characteristic $df$ (on the right) in function of $G^*$ for the first resonance frequency ($f^{(I)} = 1300$ Hz). Values are reported for the typical values expected in soft tissues, $|G^*| = 10^3 - 10^5$ Pa, corresponding to the flat region of the plots reported above in Figure 4.3.

for the first resonance frequency of the TRD device ($f^{(I)} = 1300$ Hz). The magnitude of the complex shear modulus $|G^*| = 10^3 - 10^6$ Pa varies between the typical values expected for soft internal organs to those expected for rubber materials. In Figure 4.3, the flat region corresponds to the typical values of soft tissues. Figure 4.4 shows a zoom of this region: the values expected for a soft tissue sample are in the order of magnitude of $df = 0.2 \div 0.6$ Hz and $\Delta f_{\text{res}} = -0.1 \div +0.6$ Hz. For this reason, the measurement of the TRD must be precise enough to resolve small variation of resonance frequency and damping characteristics to minimize the uncertainty of the measurements. Later in Section 4.5, issues concerning the precision and the uncertainty of the measurements executed with the TRD will be discussed in detail.
4.3 Validation of the measurement method with silicone phantoms

Before testing biological tissues, the TRD was employed with silicone and rubber phantoms that, due to a similar stiffness and viscous behavior, mimic their mechanical behavior. The main purpose of these experiments was to analyze the repeatability and assess the reliability and precision of the measurement method. To this end, the viscoelastic properties determined from the TRD tests are compared with the results obtained with wave propagation and resonance experiments on rod-like samples. The testpieces used in these tests are shown in Figure 4.5 and their main characteristics are reported in Table 4.1. We have analyzed a relatively soft elastomer (UK-IIHC/20 ShA, Kundert AG, Jona, Switzerland) and a silicone (RTV6166, GE Silicones, USA) whose stiffness properties are similar to those of soft biological tissues ($|G^*| = 10^4 \text{ } \text{ } 10^6$ Pa). The silicone used here has been previously investigated with other devices developed for soft tissue testing [65, 36] in the low frequency domain (up to 10 Hz).

Table 4.1: Materials used to validate the TRD measurement method.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Material</th>
<th>Manufacturer</th>
<th>$\rho$ [g/cm$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastomer</td>
<td>UK-IIHC/20 ShA</td>
<td>Kundert</td>
<td>1.2</td>
</tr>
<tr>
<td>Silicone 3070</td>
<td>RTV6166</td>
<td>GE Silicones</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>two-parts silicone gel</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>30% A 70% B</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 4.5: Testpieces used for the validation of the measurement method: rubber (UK-IIHC/20 ShA) and silicone (RTV6166). On the right, a rod-like sample with piezoelectric element is supported by thin wires, ready for the wave propagation tests.
TRD Tests

A cube specimen (side length 60 mm) was available for testing the elastomer and a cylindrical specimen (diameter 60 mm, height 60 mm) for the silicone. The TRD was placed in the center of a flat face. Laser interferometer measurements were performed during the experiments at the other faces of the specimen and no displacements could be detected. This was expected, due to the relatively high loss factors of these materials, and confirms that these testpieces can be modeled as a semi-infinite medium (no stationary effects due to the finite dimensions).

The tests with the TRD were conducted according to the procedure described in Section 4.2, applying a pressure $p = 0.3$ bar and making sure that no axial force was exerted on the sample, to avoid pre-deformation. The repeatability of the measurements has been investigated with multiple tests on the same testpiece. The results of the tests conducted at the first resonance frequency ($f^{(I)} = 1300$ Hz) on the elastomer are reported in Figure 4.6, in terms of the measured values of $df$ and $\Delta f_{res}$. Measurements were repeated three times in four different locations, identified by the letters A, B, C and D. The material is assumed to be homogeneous and the scatter is evaluated from the results of all locations. The resulting standard deviation for $df$ and $\Delta f_{res}$ are 0.8% and 1.3% of the respective mean values, leading to the scatter of the calculated values of $|G^*|$ and $G_2/G_1$ shown in Figure 4.7, that correspond to a Gauss distribution with a standard deviation of 1.1% and 1.0%, respectively. Similar results in terms of standard deviations were obtained with the elastomer sample at the other operative frequencies ($f^{(II)}$, $f^{(IV)}$, $f^{(V)}$). Standard deviations of up to 2% were obtained for $|G^*|$ and $G_2/G_1$ when testing the silicone sample.

Wave propagation tests

The viscoelastic properties of the synthetic materials have been measured with an alternative method to compare them with the TRD technique. Standard rheometers or quasi-static uniaxial tests cannot be used for this purpose since the strain rate in these tests is in a much lower frequency range (a maximum of 100 Hz is reached by most of the rheometers on the market) with respect to TRD. In order to achieve higher testing frequencies, wave propagation experiments have been performed on rod-like testpieces of elastomer and silicone (see Figure 4.5).

The time of flight of longitudinal waves was measured along the axis of samples, which were characterized by a square cross section of $9 \times 9$ mm and a length of 50 mm. The elastomer rod-like sample was directly extracted from the cube specimen used for the TRD experiments, while the silicone one was prepared with a mold. The experimental set-up is shown in Figure 4.8. A piezo-transducer (Ferroperm, Pz27) was glued at one extremity of the bar and was used to excite longitudinal wave pulses. Displacement due to lateral contraction was measured with a laser interferometer (Polytec OVF5000): for this purpose, the lateral surfaces of the samples have been painted with reflecting color. The laser beam was placed at different points along
4.3. Validation of the measurement method with silicone phantoms

Figure 4.6: Repeatability test on elastomer \( f^{(1)} = \SI{1300}{\text{Hz}} \). Results for multiple tests on different locations (A,B,C,D) are reported.

Figure 4.7: Gaussian distribution of \(|G^*|\) and \(G_2/G_1\) from the data of Figure 4.6. \(|G^*|\) and \(G_2/G_1\) indicate mean values and \(\sigma\) is the corresponding standard deviation.
4. Characterization of Soft Biological Tissues

![Figure 4.8: Set-up of the wave propagation experiments with a rod-like sample of synthetic material.]

the sample axis using a positioning system (Newport, IMS600) with high resolution (uni-directional repeatability 1.25 µm).

The real part of the complex longitudinal modulus, the storage modulus $E_1$, can be determined from the measured wave propagation velocity. The information on the phase angle $\delta$ (or, equivalently, on the loss modulus $E_2$) has to be inferred from an evaluation of the amplitude decay of the wave pulse along the sample. However, the quality of the measured displacement signals did not allow quantifying the displacement amplitude with sufficient precision, so that the phase angle $\delta$ could not be measured.

Narrow band longitudinal wave pulses were excited in the bar at different frequencies. For the evaluation at higher frequencies (small wavelengths) the influence of the lateral contraction has to be considered in the relationship between longitudinal wave speed and $E_1$ [26]. Experiments were conducted up to frequencies of 3500 and 1500 Hz for the elastomer and silicone sample, respectively. For higher frequencies the ratio between wavelength and polar radius of inertia of the cross section is lower than 1, so that the structural longitudinal wave model ceases to be valid [26]. This frequency range allows a comparison of the results for the first resonance frequency $f^{(I)}$ of the TRD. Results for the elastomer and silicone are reported in Figures 4.9 and 4.10. In order to validate the results at higher frequencies, the lateral dimensions of the rod sample should be reduced: this is very difficult to be done due to handling and manufacturing problems, in particular for the silicone beams.

In testing the elastomer rod-like sample, the first two longitudinal resonance modes were excited using a harmonic signal, obtaining two more data at lower fre-
4.3. Validation of the measurement method with silicone phantoms

frequencies (440 and 1040 Hz) shown in Figure 4.9. Higher resonance modes could not be excited, due to the high loss factor of the material. The elastic storage modulus of the elastomer sample was extracted at the measured resonance frequencies and the corresponding values are reported in Figure 4.9. No resonance was detected in the silicone sample due to the high material damping.

Comparison

From the wave propagation tests the longitudinal storage modulus $E_1$ has been determined. Incompressibility has been assumed for these materials [88], thus $\nu = 0.5$, leading to:

$$G_1 = \frac{E_1}{2(1 + \nu)} = \frac{E_1}{3}$$  \hspace{1cm} (4.1)

Using Equation 4.1, the corresponding values of $E_1$ were calculated from the TRD measurements of $G_1$ to enable a direct comparison with the wave propagation experiments, shown in Figures 4.9 and 4.10. An increasing trend of $E_1$ with frequency is shown from the results of both techniques, while the values found on silicone at low frequency agree with the results found in the literature with the ROSA − 2 and TeMPeST − 1D devices by Kalanovic et al. [36] on the same type of silicone. For 1300 Hz (the first resonance frequency of the TRD) the results agree to a great extent: the values differ by only 5% and 16% for the elastomer and silicone, respectively. Differences might be attributed to experimental errors, but the larger

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure4.9.png}
\caption{Characterization of the elastomer (UK-IIHC/20 ShA): comparison between results obtained with wave propagation, resonance tests, and TRD.}
\end{figure}
4. Characterization of Soft Biological Tissues

Figure 4.10: Characterization of the silicone (RTV6166): comparison between results obtained with wave propagation and TRD. Low frequency results obtained with the devices ROSA – 2 and TeMPeST – 1D by Kalanovic et al. [36] are reported for comparison.

discrepancy for silicon might be caused by the fact that the sample was not extracted from the same specimen of the TRD tests but prepared in a subsequent step: although the silicon gel components were mixed in same proportion and according to the same procedure, slight differences in the gel composition cannot be excluded.

4.3.1 The Truth Cube 2

In the previous section it was shown how the measurement technique with the TRD can be considered reliable in testing soft materials ($|G^*| = 10^3 - 10^6$Pa). Most of the devices used to measure the mechanical properties of biological tissues show results that are difficult to compare, due to the different methodologies and testing procedures used. To this end, a common project involving three different research groups (the Institute of Mechanical Systems, ETH Zurich; the CIMIT Simulation Group, Massachusetts General Hospital, Boston, USA; the Department of Health Technology and Informatics of the Hong Kong Polytechnic University, Hong Kong) was started in 2004 to propose a system for assessing the reliability of the different devices and techniques developed for testing biological tissues.

Toward this goal, a silicone phantom (called Truth Cube 2, or TC2) was created. The TC2 is a cylindrical phantom made of silicone gel (Ecoflex 0030, Smooth-on Silicons, USA), Figure 4.11, with stiffness properties comparable to those of soft biological tissues, and shows hyperelastic response up to relatively large deformations. The mechanical properties of the TC2 are stable over time, thus allowing
4.3. Validation of the measurement method with silicone phantoms

Figure 4.11: Truth Cube 2 (TC2) phantom.

direct comparison of the results obtained in subsequent investigations in different laboratories. Following previous work by Kerdok et al.[38] on a similar phantom, barium markers were embedded inside the cylinder to enable CT-scans (Computed Tomography scans) measurement of the deformation field.

The TC2 was tested using the different devices used in the three laboratories mentioned above. The results, comparing constitutive model parameters determined by each method, are an indicator of the accuracy of each approach. Independent tests were carried out on the same TC2 phantom with:

1- the Aspiration Device (later shown in Figure 4.22) at the Institute of Mechanical Systems, ETH Zurich [37, 53, 61],

2- the Torsional Resonator Device TRD described in this work,

3- the Minibird, a spatial locating sensor based on tissue indentation [101] at the Hong Kong Polytechnic University,

4- the TeMPeST-1D, a device based on tissue indentation [65] at the CIMIT Simulation Group,

5- a standard shear rheometer (AR2000 rheometer, TA Instruments) at the CIMIT Simulation Group,

6- a standard uni-axial test (Zwick/Roll 1456 tension-compression testing machine) at the Institute of Mechanical Systems, ETH Zurich.
Figure 4.12 shows a complete picture of the mechanical properties of the TC2 phantom in a wide frequency range. On the left, the results obtained with the quasi-static tests (1, 3, 4, 6) are reported. A mean value of the Young’s modulus of 27.5 kPa was obtained, showing good agreement between the different methods considered. The Tempest-1D seems to overestimate the elastic modulus. On the right, the results obtained in the frequency domain (2, 4, 5) are reported. An increase in the stiffness properties (similar to what observed in Figure 4.9 and 4.10) is registered. The TRD returns the results located at the right of Figure 4.12, in the high frequency range. The increase of the stiffness properties of the materials at high frequencies is a common phenomenon also found by other authors [21]. Further results and comments on this comparison are discussed in Hollenstein et al. [89]. The values of the material parameters determined in this investigation can also be used as a reference by other measurements methods that aim at finding mechanical properties of soft materials for validation purposes.

**Figure 4.12:** Results obtained on the TC2 with quasi-static methods (1, 3, 4 and 6 reported on the left) and dynamic methods (2, 4 and 5, on the right).
4.4 Results on soft biological tissues

The TRD technique was applied to test bovine organs ex vivo. Adult bovine liver, kidney, and uterus samples, obtained from the local abattoir, were tested at ambient temperature. The intact bovine organs were obtained from the slaughterhouse immediately following animal euthanasia. The organs were transported inside a coolbox and kept moist, wrapped in a physiological saline soaked surgical cloth, to prevent dehydration of the surface. The tests presented here were executed when the tissue samples had already reached a stable ambient temperature of 22-24°C.

In contrast to the synthetic materials previously examined, soft tissues are inhomogeneous, can be composed of different layers (e.g. membranes that cover internal organs), are highly influenced by environmental effects (e.g. dehydration, temperature) and do not have a regular geometry. The consequences of these characteristics on the TRD experiments are analyzed with the present test series.

4.4.1 Repeatability

Figure 4.14 shows the results of the measurements on bovine liver in terms of $df$ and $\Delta f_{res}$. Measurements were performed at the external surface of the organ, so that the TRD was in contact with the external membrane (called capsule) that covers the liver. The first eigenfrequency of the TRD ($f^{(I)} = 1300$ Hz) was considered in this analysis. A series of measurements was performed on the same organ at different locations, identified by the letters A, B, C, D, E, and F as shown in Figure 4.13. Three measurements have been performed within short time intervals at each location.

Figure 4.13: A bovine liver sample used in the measurement with the TRD, with letters indicating the different locations of the tests.
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Figure 4.14: Repeatability tests on bovine liver (with capsule). Results for multiple tests at different locations (A-F) are reported.

Figure 4.15: Gaussian distribution of $|G^*|$ and $G_2/G_1$, and point-normalized data ($|G^*|$)$_{NORM}$ and $(G_2/G_1)$_{NORM} from the data of Figure 4.14. Mean values and corresponding standard deviation $\sigma$ are reported.
The damping characteristic $df$ is in the order of 0.55 Hz, with a standard deviation over all measurements of 12%, corresponding to a quality factor $Q$ in the order of 2400, ten times smaller than in the calibration run (index of good sensibility of the device). Very small variations are observed for the resonance frequency $\Delta f_{res}$, about less than 0.1 Hz. As a consequence, the standard deviation over all measurements is over 100% for $\Delta f_{res}$. The corresponding scatter of the calculated values of $|G^*|$ and $G_2/G_1$ is shown in Figure 4.15a and 4.15b, that corresponds to a Gauss distribution with standard deviation of 29% and 42%, respectively.

In order to eliminate the variability from location to location, the $|G^*|$ and $G_2/G_1$ values of each location (A, B, C, D, E, and F) were normalized with respect to their local specific averages. The standard deviation of the normalized values ($|G^*|_{NORM}$ and $(G_2/G_1)_{NORM}$ is 13% and 38% respectively, leading to the distributions shown in Figure 4.15c and 4.15d. This variability has to be attributed to the uncertainties of the experimental procedure and will be further discussed.

Figure 4.16 shows the time evolution of $|G^*|$ for TRD measurements performed at one single location at 1300 Hz over a time range of 4 hours. The organ was exposed to air in ambient conditions. The strong increase in stiffness is caused by tissue alterations due to dehydration and oxidation.

**Figure 4.16:** Time evolution of $|G^*|$ on bovine liver (with capsule) due to dehydration.
4. Characterization of Soft Biological Tissues

4.4.2 Experiments with different bovine organs

In addition to the experiments performed at the external surface of the liver (referred to as liver with capsule), measurements at a sectioned surface of the organ (referred to as liver parenchyma) were carried out. Furthermore, measurements at the external surface of kidney and uterus were also performed. Table 4.2 shows mean values ($|G^*|$ and $G_2/G_1$) and standard deviations ($\sigma_{|G^*|}$ and $\sigma_{G_2/G_1}$) of the measurements carried out at 1300 Hz. Five tests (at five different locations) were performed on the same organ.

In Table 4.2, the values of $|G^*|$ obtained for liver parenchyma, kidney and uterus are similar, in contrast to the results obtained in the tests executed at the external surface of the liver (liver with capsule), which are at least three times higher. A stiffening effect brought by the presence of the external covering membrane is supposed and will be further investigated and commented in Section 4.4.4.

The estimated material loss factor $\bar{\eta} = G_2/G_1$ varies between 0.470 and 0.597 for the liver (with capsule), liver parenchyma and kidney, while it reaches a high value for the uterus ($G_2/G_1 = 2.856$). This strong viscous behavior for the uterus reflects the presence of a highly viscous mucus on its surface that affects sensibly the results, also in terms of the scatter. More comments on the scatter of the results and on the error sources will be presented later in Section 4.5.

**Table 4.2:** TRD results on bovine organs at 1300 Hz

| Sample              | $|G^*|$ [Pa] | $\sigma_{|G^*|}$ [Pa] | $G_2/G_1$  | $\sigma_{G_2/G_1}$ |
|---------------------|------------|----------------------|------------|-------------------|
| Liver (with capsule)| 19942      | 5743                 | 0.597      | 0.253             |
| Liver (parenchyma)  | 6828       | 1183                 | 0.470      | 0.142             |
| Kidney              | 6767       | 1076                 | 0.484      | 0.113             |
| Uterus              | 5912       | 2079                 | 2.856      | 0.493             |

Further tests were conducted on bovine livers extracted from five different animals, executing the measurements with the capsule. In Figure 4.17, the results in terms of $|G^*|$ obtained at 1.3 kHz are compared. The error bars correspond to the standard deviation $\sigma_{|G^*|}$ from 12 measurements at different locations on each organ. In general, the variability between different organs is of the same order of magnitude of the variability within one single organ.
4.4. Results on soft biological tissues

![Graph showing variability of |G*| measurements of bovine liver (with capsule).](image)

**Figure 4.17:** Variability of the $|G^*|$ measurements of bovine liver (with capsule). Organs obtained from five different animals, tested at 1300 Hz.

4.4.3 Frequency dependence

Performing experiments at the different eigenfrequencies of the resonator, the frequency dependence of the material behavior can be investigated. Results are reported for the first, the third, the fourth and the fifth eigenfrequency of the TRD.

The measured values of the shear modulus $|G^*|$ on bovine liver are shown in Figure 4.18, which includes the results obtained from tests of the liver with capsule and liver parenchyma. The comparison highlights the strong stiffening effect of the capsule over the entire frequency range considered. Five test (at five different locations) were performed on the same organ and the corresponding variability is indicated in Figure 4.18 by error bars, corresponding to an interval of $\pm \sigma$ (where $\sigma$ indicates the standard deviation).

In Figure 4.19, the results for kidney and uterus are reported. The larger scatter of uterus measurements (mainly due to the difficulties encountered in testing the surface of this organ widely covered by mucus) is confirmed also at higher frequencies. Higher repeatability was observed when testing kidney.

The values of the phase angle $\delta = \arctan \left( \frac{G_2}{G_1} \right)$ measured during the tests, in all the cases analyzed, were unstable and highly affected by a strong scatter, that could reach up to 100% of the mean value. For this reason, no figure is presented in this thesis upon the frequency dependence of the phase angle $\delta$ in biological tissues (with the exception of Figure 4.24, that is presented later). The high uncertainty found in this parameter is related to an intrinsic precision problem of the TRD measurement technique (precision required for $\Delta f_{res}$ and is discussed in detail in Section 4.5).
4. Characterization of Soft Biological Tissues

Figure 4.18: Frequency dependence of the magnitude of the complex shear modulus $|G^*|$ of bovine liver: measurements executed at the external surface of the liver (with capsule) and at a sectioned surface of the organ (parenchyma). 5 tests were carried out at different locations for each resonance frequency. The error bars correspond to an interval ± standard deviation.

Figure 4.19: Frequency dependence of the magnitude of the complex shear modulus $|G^*|$ of bovine kidney and uterus. 5 tests were carried out at different locations for each resonance frequency. The error bars correspond to an interval ± standard deviation.
4.4.4 Bi-layer model of the liver

The importance of the capsule in modeling the mechanical behavior of internal organs has been investigated considering the example of bovine liver. The results presented in Figure 4.18 have been obtained under the assumption of homogenous material (using the analytical solution for a half-space medium). The values obtained by performing a measurement with the TRD on the capsule, without removing this thin membrane, do not represent the properties of the parenchyma (a clear overestimation is evident from Figure 4.18), nor the properties of the capsule alone.

As mentioned in Chapter 3, a global characterization of a bi-layered material is not possible using a unique TRD measurement, due to the many parameters that have to be assessed ($G_1^*$, complex shear module of the capsule, considered as layer 1, and $G_2^*$, complex shear module of the parenchyma, layer 2). However, it is possible to evaluate the impact of a stiffer membrane on the global properties of the bulk organ by performing two experiments, one with the capsule and one without it, measuring the parenchyma after having removed the capsule. A test executed on the capsule alone (in form of a thin membrane, after removal from the parenchyma) with the TRD would also be an alternative choice. Unfortunately, due to the difficulties encountered in peeling off the capsule from the parenchyma, (pre-stressing the material) and to reproduce the initial pre-deformed state in the experiments, a reliable test could not be performed.

In Section 3.2.2, it was discussed how the presence of a layer over a half-space influences the results of a dynamic torsion test. This model is now applied to the

\begin{figure}[h]
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\includegraphics[width=\textwidth]{image.png}
\caption{The membrane that covers a bovine liver (capsule) is peeled off from the parenchyma using fingers and surgical forceps.}
\end{figure}
characterization of bovine liver, assuming that the capsule has a constant thickness $q_1$ and is perfectly bonded to the liver parenchyma, which is treated as a half-space. The capsule was peeled off from the liver using fingers and surgical forceps, as shown in Figure 4.20. The thickness of the capsula was assessed by averaging several measurements taken at random locations using a micrometer caliper, resulting into a value of $q_1 = 95 \pm 15 \mu m$. By knowing the thickness of the membrane, its mechanical properties $G_1^*$ can be searched for, using the value of $G_2^*$ (complex shear module of the layer 2, in this case the parenchyma) estimated after having removed the first layer. The estimated value of $G_1^*$ represents the shear modulus of the capsule corresponding to the pre-stretch level typical of in vivo condition.

The results are reported in Figure 4.21, where the complex shear modulus of the capsule (in terms of its magnitude $|G_1^*|$) is compared to the values measured for the internal parenchyma ($|G_2^*|$), and to the apparent value obtained with a homogenous material assumption ($|G_{app}^*|$), using the results of tests executed on the intact organ. These results show how stiff this membrane is, almost one order of magnitude stiffer than the internal parenchyma, at each frequency of test. The capsule has a stiffness varying from 125 kPa, at 1.3 kHz, up to 657 kPa at 12 kHz.

A comparison to similar results obtained from quasi-static and uniaxial elongation tests recently presented by Hollenstein et al. [32] points out a general agreement with the TRD tests. In uniaxial tests executed on the capsule alone after removal from the parenchyma, Hollenstein et al. [32] found that the elastic parameters of the capsule were considerably higher, at least 2 to 3 order of magnitudes, than those of

![Figure 4.21: Results of the bi-layer model for bovine liver capsule. The values obtained from a homogenous model of the organ (measurement on the capsule and measurement after removal of the capsule) are reported for comparison.](image-url)
the parenchyma. In their work, it was also pointed out that the presence of a capsule influences to a great extent the outcomes of the tests, if one aims at estimating the properties of the parenchyma with an aspiration test.

However, a discrepancy in the quantitative values estimated by the TRD test and by Hollenstein et al. [32] for the shear modulus $|G_1^*|$ can be evidenced. In their experiments, a linearized shear modulus (valid for infinitesimal strains) of 366 kPa was found, that is considerably higher than the value returned by the TRD at the first (lowest) resonance frequency ($|G_1^*|=125$ kPa, see Figure 4.21). This discrepancy may be due to: (i) difficult evaluation of the mechanical properties of the capsule, which exhibits a highly nonlinear behavior depending on the strain level: the elastic properties are highly strain-dependent, and the methodology proposed in [32] cannot be precise enough at infinitesimal strains to extract a linearized elastic modulus, due to the nature of the experiments executed at large strains, (ii) preconditioning of the samples during the removal from the parenchyma, that might affect the fiber orientation in the samples resulting in material hardening, (iii) inexact evaluation of the thickness $q$ used to extract the results from the TRD, which is difficult to evaluate precisely and can vary depending on the position, within the same organ, (iv) anisotropy of the capsule, which mainly consists of fibers, that can exhibit a response to infinitesimal shear strains (TRD tests) different than what observed in uniaxial or equibiaxial test (as reported in [32]), depending on fibers alignment.

Further investigation on this topic is required to shed light on these discrepancies, testing a higher number of organs characterized by different thickness of the capsule and investigating the effect of anisotropy. Future application of the TRD device in the characterization of soft biological tissues during in vivo tests must consider the importance of this stiffening effect brought by the capsule. Due to the influence of this stiffer membrane, the evaluation of the liver as a homogenous tissue leads to an overestimation of the properties of the parenchyma by a factor of 2 to 3.

**4.4.5 Combined experiments with the Aspiration Device**

The TRD can be employed together with the Aspiration Device [91, 60], in order to obtain a full characterization of soft biological tissues, giving an insight of the mechanical properties over a wide frequency range. The Aspiration Device is shown in Figure 4.22 along with a scheme of its working principle. This device performs quasi-static aspiration tests on soft material samples, whose properties are characterized with hyperelastic material laws in large deformations (up to 30% nominal strain). In Appendix B.2, further details on the experimental procedure and on the hyperelastic material laws used to model soft biological tissues are reported.

The results obtained with this quasi-static technique can be compared to those obtained with the TRD in the high frequency domain. From the constitutive model determined with the aspiration tests (a fifth order reduced polynomial form hyperelastic model with viscoelasticity included through Prony series [22]), a linearized shear relaxation modulus $G(t)$ is calculated (see Appendix B.2). The frequency...
dependent complex shear modulus $G^*$ is calculated with the relation:

$$G^*(\omega) = G(0) + \int_0^\infty e^{-j\omega \tau} \dot{G}(\tau) d\tau$$

(4.2)

A series of combined experiments on an adult bovine liver were carried out with the Aspiration Device (quasi-static tests) and with the TRD (dynamic tests) in order to compare the results of these two techniques in the frequency domain. Experiments were performed with the two techniques on the same location of an intact bovine liver (without removing the capsule) within a short time interval. The combination of the two methods yields a characterization of the material response over a wide range of frequencies.

In Figure 4.23, the magnitude of the shear modulus $|G^*|$ is compared, while in Figure 4.24 the phase angle $\delta$ is reported. The error bars in these two figures indicate: (i) for the Aspiration Device, the variability due to the contact force [60] and the variation of the shear modulus in the frequency range 0.001-0.1 Hz, (ii) for the TRD, the standard deviations of 5 measurements executed at each eigenfrequency, right after the execution of the aspiration tests. A significant frequency dependence of $|G^*|$ is demonstrated by these measurements. A linear dependence of the shear modulus upon the frequency can be assumed from Figure 4.24.

A comparison of the viscous behavior registered by the two methods is difficult. The Aspiration Device returns a value of $\delta=0.11-0.23$, that is considerably smaller than the mean values returned by the TRD. The scatter of the results obtained with the TRD in the phase angle is too high to extract a precise trend out of these data. A constant $Q$ factor model, as suggested by Fung [22] for biological tissues (meaning that the hysteresis in the material is independent from the strain rate), would require a stable value of $\eta = \tan \delta$ that cannot be confirmed by these tests. Further investigations must be carried out to increase the precision of the phase angle estimation with the TRD, a common problem in all the tests performed on biological tissues discussed in the next section.
4.4. Results on soft biological tissues

**Figure 4.23:** Results from quasi-static aspiration experiments [61] and TRD tests: magnitude of the complex shear modulus $|G^*|$.  

**Figure 4.24:** Results from quasi-static experiments [61] and TRD tests: phase angle $\delta$ of the complex shear modulus.
4.5 Discussion

Validation of the method

The reliability of the experimental procedure of the TRD and the analytical model are supported by the agreement of the results from TRD measurements and wave propagation experiments, as well as by the high repeatability of the measurements with synthetic materials. A validation of the method over a larger range of frequencies and material properties requires dynamic experiments that are not easily performed with soft, highly damped materials. Several difficulties were encountered when performing wave propagation experiments at high frequencies related to (i) sample preparation, (ii) problems in handling very soft slender rod-like samples, (iii) strong amplitude decay, in particular at high frequencies, leading to poor accuracy in displacement measurements. These problems highlighted the advantages of the TRD technique, for which no specific sample geometry is required, testing and data analysis is simple, fast and accurate, and rheological properties can be easily obtained for frequencies up to 12 kHz.

Multiple experiments with synthetic materials with $|G^*|$ values larger than $10^5$ Pa led to very high repeatability, with standard deviations as low as 1% with respect to the mean values of $|G^*|$ and $G_2/G_1$. The repeatability was significantly less in the experiments with soft biological tissue. The relatively large scatter obtained from measurements on liver at different locations (with standard deviation of 29% and 42% of the mean values of $|G^*|$ and $G_2/G_1$) is not surprising when compared to published experimental data on biological tissue (see e.g. [12, 61, 84]). One important influential factor of this variability is the non-uniformity of the tissue, so that different responses are obtained at different locations in one organ.

The variability of the normalized $(|G^*|)_{\text{NORM}}$ and $(G_2/G_1)_{\text{NORM}}$ values for liver (Figure 4.15c and 4.15d) however is due to the uncertainty of the experimental procedure. The corresponding standard deviations for $(|G^*|)_{\text{NORM}}$ and $(G_2/G_1)_{\text{NORM}}$ are 13% and 38% respectively. Larger variability of $G_2/G_1$ is due to the fact that both $df$ and $\Delta f_{\text{res}}$ are more sensitive to variations in $|G^*|$ than to variations in $G_2/G_1$, see Figure 4.4, so that uncertainties in measurement of $df$ and $\Delta f_{\text{res}}$ have more influence on $G_2/G_1$.

Uncertainties and critical factors in the procedure

Larger scatter in both $|G^*|$ and $G_2/G_1$ of liver with respect to synthetic materials can be mainly attributed to the uncertainties in the determination of $\Delta f_{\text{res}}$. As shown in Figure 4.4, $\Delta f_{\text{res}}$ values of less than 0.1 Hz are expected for a material with $|G^*|$ lower than 50000 Pa (as for the soft internal organs considered).

The phase stabilization loop allows to determine the resonance frequency with an accuracy of $10^{-6}$ in the present configuration (corresponding to about 0.001 Hz for the first resonance frequency $f^{(1)} = 1300$ Hz). The resonance frequency of the torsional resonator however is subjected to a larger variability, mainly due to the influence of temperature. At 1300 Hz the standard deviation of a series of
Figure 4.25: Error evaluation on the parameters $|G^*|$ and $G_2/G_1$ due to an uncertainty of the resonance frequency measurement $\epsilon(\Delta f_{\text{res}}) = \pm 0.02$ Hz. A value of $G_2/G_1 = 0.5$ has been assumed.

calibration runs (without contact with the tissue sample) is approximately 0.02 Hz. This variability corresponds to temperature fluctuations in the order of 0.1°C. Using the analytical model and assuming a material with $G_2/G_1 = 0.5$, the error in the determination of $|G^*|$ and $G_2/G_1$ due to a variation of the resonance frequency of $\epsilon(\Delta f_{\text{res}}) = \pm 0.02$ Hz can be calculated. The resulting errors are shown in Figure 4.25 for different values of $|G^*|$. The curves show that: (i) the error in $G_2/G_1$ is always larger than the error in $|G^*|$. (ii) For values of $|G^*|$ in the range of $10^5 - 10^6$ Pa (synthetic materials) errors are small; (iii) errors increase with lower values of $|G^*|$. For $|G^*|$ in the range of $10^4$ Pa (as in the case of soft internal organs) errors are in the range of 6% and 45% for $|G^*|$ and $G_2/G_1$, respectively. This error source represent therefore a significant contribution to the scatter of $(|G^*|)_{\text{NORM}}$ and $(G_2/G_1)_{\text{NORM}}$ obtained with the organs samples.

In addition to the above considerations, larger uncertainties affect the measurement when testing biological tissue with respect to synthetic materials, and are due to: (i) possible dependence of the mechanical response on the loading history (non-linear viscoelastic behavior); (ii) the irregular shape of the sample surface which influences the contact area, with localized normal forces arising that are possibly not detected by the balance reading; (iii) the tissue alterations due to dehydration leading to change of properties with time (see Figure 4.16). The observed tissue alteration with time highlights the difficulties related to ex vivo biomechanical experiments, with the biological tissue changing its mechanical response when extracted from the natural biochemical environment.

The results of Figures 4.18 and 4.19 show an increase of $|G^*|$ with frequency.
This is in agreement with the results on soft internal organs described for lower frequencies by Nasseri et al. [59], 0.01-10Hz, by Kruse et al. [42] and Manduca et al. [51], 50-300Hz. A frequency characterization of the loss factor of soft tissues, in terms of the ratio $G_2/G_1$, was not presented in this work due to the high scatter registered. As previously pointed out, the uncertainty of the measurement of the resonance frequency shift $\epsilon(\Delta f_{res})$, strongly affects the estimation of this parameter.

Figure 4.18 allows a comparison between a measurement of $|G^*|$ obtained at the external surface with one obtained testing the parenchyma. The difference is significant and is due to the relatively stiff membrane (capsule), which influences to a great extent the results of the test. By using the analytical model of a bi-layered material, the mechanical properties of the capsule can be determined. The complex shear modulus of the capsule alone ($G_1^*$) was obtained from a combination of the results of surface and liver interior experiments. These results showed a value of the complex shear modulus ($|G_1^*|$) which is at least one order of magnitude higher than the one obtained testing liver parenchyma alone ($|G_2^*|$), for the frequencies and strain ranges considered here (1-12 kHz, $\gamma \approx 0.1\%$). Neglecting the influence of the capsule leads to a significant overestimation of the properties of the internal parenchyma. This demonstrates the importance of a correct modeling of the capsule for (i) simulation of organ behavior, and (ii) interpretation of experimental data for diagnostic purposes.

4.6 Conclusions and outlook

In this chapter, the dynamic torsion test was applied to soft biological tissue, performing experiments with the TRD on bovine internal organs during ex vivo experiments. The measurement technique was first validated through comparative measurements on silicone and rubber materials. The results of tests on bovine organs demonstrated the suitability of this experimental procedure for the characterization of biological tissues.

The repeatability tests have shown that high accuracy can be expected from experiments with materials in the range $|G^*| \sim 10^5 - 10^6$ Pa. Thus, this technique can be useful for characterizing soft polymers at frequencies up to 12 kHz with the advantage of simple and fast testing and data analysis.

Larger scatter has been observed for biological tissue, being the stability of the resonance frequency the main error source. Future work must focus on the reduction in the variability of the TRD resonance frequency. The resulting uncertainties in the rheological properties estimated, however, seem to be acceptable when compared to the scatter in the results of other testing methods and to the inherent variability of mechanical properties within soft organs.

Insight in the mechanical response of soft biological tissue is provided by the experiments presented here: (i) measured values of $|G^*|$ are in the range of $10^3 - 10^5$ Pa; (ii) for all organs $|G^*|$ increases with frequency; (iii) the capsule has a significant stiffening effect on the mechanical response at the liver surface; (iv) dehydration
leads to significant changes in tissue rheological properties.

One significant limitation of the present experiment and analysis technique is represented by the fact that characterization of the rheological properties of biological or synthetic tissues can be obtained only for homogeneous and isotropic samples. An extension of the present modeling approach should be planned to investigate the influence of anisotropy and inhomogeneity on the interaction between sample and resonator. The finite element approach for the solution of the torsional vibrations problem, presented in Chapter 3, could be used to this end.

Applications of the TRD technique in diagnostics can be explored in future work. No damage is caused by the experiment so that this method could be applied in vivo on human organs: during surgical interventions or organ inspections a local measurement of rheological properties could help distinguishing between healthy and unhealthy tissue. Possible developments toward in vivo applications include: (i) a support and positioning system for the TRD device for use in the operation room; (ii) a load cell to ensure zero normal force during measurements; (iii) a computer algorithm for on-line extraction of viscoelastic parameters from the measured values of $\Delta f_{res}$ and $df$. 
4. Characterization of Soft Biological Tissues
Chapter 5

Characterization of Synthetic Materials

The dynamic torsion test presented in this thesis was developed for the main purpose of characterizing soft biological tissues. The device conception and the analytical and finite element models described in Chapter 2 and 3 can also be applied in testing other materials, in order to characterize their viscoelastic behavior in the high frequency domain. This chapter presents the results obtained from tests on bituminous binders and on electroactive polymers, showing the effectiveness of this measurement method in different application fields. Results obtained with the HFTR in laboratory and field tests in order to investigate the aging phenomenon of asphalt mixtures are presented in this chapter. The mechanical properties of electroactive polymers, in form of thin pre-stretched membrane samples, were also characterized with combined experiments with both devices, HFTR and TRD, giving some insight in the rheological properties of these materials at high frequencies.

5.1 Introduction

The dynamic torsion test presented in this thesis has been successfully applied in testing silicone and rubber samples in Chapter 4 for validation purposes. In Section 4.5, it has been pointed out that the sensitivity of the dynamic torsion test is related to the characteristics of the materials that have to be measured. A higher sensitivity of the measurement was observed for stiffer materials: for the TRD, $|G^*| > 10^5 \text{Pa}$ as reported in Figure 4.25, guarantees that fluctuations of the resonance frequency in the order of 0.02 Hz do not influence the estimated complex shear modulus more than 5%. In this chapter, two different synthetic materials, characterized by a strong viscoelastic behavior (loss factors $\eta > 0.5$), are analyzed with the dynamic torsion
5. Characterization of Synthetic Materials

test using both HFTR and TRD: (i) bituminous binders and (ii) electroactive polymers (EAPs). The results presented here confirm the reliability of the measurement method of the dynamic torsion test and provide useful information about the high frequency rheological behavior of (i) and (ii).

Furthermore, the results presented in this chapter demonstrate that the procedure of the dynamic torsion test can be applied to: (a) membrane-like samples, (b) layered samples, whose mechanical properties are dependent upon the depth, and (c) tests with sensors partially embedded into material samples (lateral contact).

**Bituminous binders**

Most road pavements are constructed using a mixture of mineral aggregates and a binder, blended at high temperature (135-163°C) [15], which are mixed together, laid down in layers and compacted. The performance of a road pavement is controlled mainly by the properties of the binder, the only deformable component, which forms a continuous matrix [46]. Bitumen, the most popular asphalt binder, is the highly viscous residue of crude oil, obtained by removing most of its volatile components. In the last few years, the increasing demand on road performance due to higher traffic volumes and vehicles loads has favored the use of polymer-modified bitumen, because polymer addition imparts enhanced service properties to the pavement over a wide range of temperatures, reducing the frequency of maintenance required on highways. Copolymers, such as styrene-butadiene-styrene (SBS) triblock copolymers [21], have been proved very effective modifiers for base bitumen [13]. Improvement in the resistance to rutting (permanent deformation of the pavement in the form of ruts or corrugations) and thermal cracking (fracture of the pavement due to the lack of flexibility, at low temperatures) has been demonstrated by the addition of polymers to the base bitumen.

The mechanical behavior of base bituminous binders (or so called straight run bituminous binders) and of polymer modified (PMB) bituminous binders are influenced by prolonged exposure to air, temperature and environmental effects under their typical working conditions, which lead to significant deterioration of road performance. The aging process in a pavement consists of continuous degradation and transformation of the components in bituminous binders under production and field conditions. Bitumen aging takes place in two stages: short-term and long-term [49]. Short-term aging occurs during the production, storage, laying, and compaction of asphalt mixtures. Long-term aging is a process mainly caused by the exposure of binders to air, where bitumen oxidation occurs and temperature plays an important role. The continuous hardening and embrittlement of binders typically observed during aging leads to a progressive deterioration of asphalt pavements, which has direct consequences in road safety and maintenance, resulting in major economic loss.

Aging can be studied in laboratories, through so called accelerated procedures, or directly during field tests in the typical operative conditions observed on roads. Aging of bituminous binders is commonly simulated in laboratory tests using vari-
ous accelerated procedures, like the rolling thin film oven test RTFOT and thin film oven test TFOT, or the pressure aging vessel PAV [25, 34]. In these tests, bitumen aging is accelerated by increasing temperature, decreasing bitumen film thickness, increasing oxygen pressure, or applying various combinations of these factors. After these treatments, samples of thin films are usually mixed together in a homogenizing process and the resulting mixture is further used for material properties testing using DMA (e.g. [33, 48, 52, 62, 76, 97]) quantifying the changes in the elastic and viscous properties of the asphalt binders over time. These laboratory aging procedures aim to simulate the environmental conditions observed during the manufacture and construction of the pavements and later on the field during their service life. However, due to the difficulties of simulating environmental conditions during production and service life, a direct relationship between laboratory and field aging is difficult to obtain. The evaluation of bitumen aging through field tests can provide interesting information on the time evolution of the rheological properties of the pavements. However, this is not an easy task due to the experimental problems that arise in measuring the rheological properties directly on the field, since most common rheometers cannot be employed.

Aging in bituminous binders can be investigated with the dynamic torsion test and is the subject of the present work. The particular features of this measurement technique lead to a series of advantages in the investigation of bitumen aging: (i) the devices are portable, enabling in-situ characterization of the mechanical properties of the binders; (ii) the devices are robust in environment where traffic-induced low frequencies can affect the measurements, operating in the high frequency range, this being useful for field tests; (iii) due to their relatively small size, they can be inserted in oven and pressure aging vessels to characterize the mechanical properties during the execution of a test. To this end, two variants of the HFTR (whose description was reported in Chapter 2) were used, one for laboratory tests and one for field tests.

The aging of a straight run bituminous binder 180/200 was studied in laboratory using the HFTR, quantifying the rheological changes that the material undergoes during exposure to air pressure and temperature in terms of variation of the complex shear modulus $G^*$. A comparison with results obtained with a standard dynamic shear rheometer at low frequencies on the same straight run bitumen is presented for validation purposes. The aging of bituminous binders is then studied directly on the field, interpreting the results obtained with the HFTR for field tests on an asphaltic plug joint located on a highway bridge over three consecutive years, monitoring the changes in the viscoelastic behavior of the material (an elastomeric SBS, styrene-butadiene-styrene [13], polymer modified bitumen) over three years of field tests, exposed to atmospheric conditions. The results of these aging test, already presented by Poulidakos et al. [9] in terms of changes in equivalent stiffness and damping coefficients, are interpreted here quantitatively in terms of the variation in the material complex shear modulus $G^*$ as a function of temperature and time of exposure, giving therefore some insight into the rheological properties of polymer modified bitumen during the aging process.
Electroactive polymers

Electroactive polymers (EAPs) are functional materials that can be used as actuators in adaptive structures, in particular when large deformations are required. EAPs transform electric energy directly into mechanical work and produce large strains, in the order of 10%-30%. Dielectric elastomer actuators are a category of EAPs which were shown to provide excellent overall performance [7], combining large elongation, high energy density, good efficiency and high speed of response. Examples of applications [66, 67] of dielectric elastomers as actuators include mobile mini- and microrobots, micropumps and microvalves, micro air vehicles, disk drives, prosthetic devices and flat panel loudspeakers.

Due to the different types of applications for which these materials are employed, the response of dielectric elastomers to a variety of loads must be considered: the strain can reach levels of about 500%, with strain rates in the kHz region for acoustic applications and vibration isolation. Dielectric elastomers are usually tested with classic uniaxial machines, performing quasi-static tensile and relaxation tests at different values of the nominal strain [7, 40]. Their behavior in large deformations can be modeled through hyperelastic material laws normally proposed for elastomers and rubbers [95, 96]. Quasi-linear viscoelastic theory [22] can be assumed to reproduce the time dependent behavior typically observed, as proposed by Wissler and Mazza [93]. Dynamic mechanical analysis (DMA) is used to characterize the frequency dependent behavior for the frequency range 0.001-100 Hz [87].

The dynamic torsion test can contribute to extend the knowledge of the viscoelastic behavior in dielectric elastomers up to a frequency of 12 kHz. The possibility to execute measurements at different levels of pre-strain, by testing specimens that have been pre-strained prior to the tests, can be used to obtain information on the nonlinear behavior of these materials.

A particular type of dielectric elastomer, VHB 4910, was characterized here in the high frequency domain at different pre-strain levels. Tests were executed on membrane-like specimens of VHB 4910 with the TRD and HFTR sensors. The specimens analyzed were obtained by applying different pre-strains to virgin foils of dielectric elastomers, varying from the undeformed state to a 500% nominal equibiaxial strain. The capability of the TRD and HFTR to be applied in tests of thin, membrane-like samples of elastomers is demonstrated in this chapter. A comparison with results obtained with quasi-static tests performed with uniaxial tensile machines shows that the linearized mechanical behavior predicted by the dynamic torsion test agrees to a good extent with the quasi-static findings.
5.2 Laboratory aging tests of bituminous binders

5.2.1 Dynamic mechanical analysis

Dynamic mechanical analysis (DMA) is the common technique used to measure the rheological properties of bituminous binders and asphalt mixes, essentially enabling the viscoelastic nature of the material to be characterized. The parallel-disk configuration and the rectangular bar configuration are the standard procedures considered in European standards [25, 34]. In both cases, the test specimen is subjected to an oscillatory shear strain and the harmonic functions of the resultant stress are measured. When a bituminous binder is cold and brittle it behaves like an elastic solid and its stress signal is in phase with the sinusoidal strain. At high temperatures it will approach an ideal viscous fluid behavior and the stress lags 90° behind the strain. At intermediate temperatures (20°-50°C), in the working range of bituminous binders, the material behavior is viscoelastic so that the stress can lag within a range of 0°-90° behind the strain.

Bituminous binders are usually characterized by measuring the magnitude of the complex shear modulus $|G^*|$ and its phase $\delta$ or, equivalently, the loss factor $\tan \delta = G_2/G_1$, as a function of temperature and frequency. A dynamic shear rheometer (Bohlin, DSR-50) was used in frequency- and temperature-controlled tests in order to have reference values to be compared with the HFTR for the bituminous binders analyzed.

![High Frequency Torsional Rheometer (HFTR) with stand. The vertical position of the rheometer with respect to the sample can be adjusted with a micrometer screw.](image)

**Figure 5.1:** High Frequency Torsional Rheometer (HFTR) with stand. The vertical position of the rheometer with respect to the sample can be adjusted with a micrometer screw.
5.2.2 High frequency rheometry

The viscoelastic properties of bituminous binders are investigated in the laboratory using the High Frequency Torsional Rheometer (HFTR) depicted in Figure 5.1. The design details of this sensor have been reported in Section 2.3.2. The sensor can be simply laid on the surface of a specimen or partially lowered into it, by controlling the vertical displacement through a travel stage (micrometer screw). In this way, the material can be characterized at different depths.

From the measured values of $\Delta f_{res}$ and $df$, the complex shear modulus $G^*$ of the material in contact with the sensor can be extracted by means of the analytical or finite element models presented in Chapter 3. In this case, the finite element solution can be of particular advantage, since the half-space assumption, required in the analytical solutions, may not be applied in case of specimens of small size or hard materials. Moreover, the influence of a lateral contact on the resonator (when the sensor is partially lowered into the material) can be more effectively studied with a numerical approach rather than with an analytical solution. In Figure 5.2, the FE model of the HFTR sensor in contact with a bituminous material sample is shown.

![Figure 5.2: Full 3D finite element model of the HFTR sensor (a) and model of an angular section (b) with cyclic symmetry boundary conditions. Colors represent the magnitude of the displacement field $|u_{\varphi}|$. The displacement is normalized with respect to the displacement at the tip of the resonator.](image)

5.2.3 Material and samples preparation

The viscoelastic characteristics of the straight run penetration graded bitumen 180/200 (CTW-Strassenbaustoffe Sika AG, Switzerland) have been analyzed using the HFTR during accelerated aging in laboratory. Prior to the aging experiments, standard tests on the 180/200 binder have been carried out showing a density $\rho = 1039 \text{ kg/m}^3$, a value of $16.8 \text{ mm}^{-1}$ for the needle penetration test at $25^\circ\text{C}$ and a
5.2. Laboratory aging tests of bituminous binders

ring and ball softening point of 43.2°C. Cylindrical samples of this binder (diameter 50 mm, height 20 mm, weight 35 g) were prepared inside glass container (see Figure 5.7). The samples were taken from the containers received from the suppliers using heated hook and knife. The bitumen itself was not heated in order to avoid any modification of the samples before testing. The dimensions of these specimens are big enough for the assumption of viscoelastic half-space necessary in the analytical model used for the extraction of the mechanical parameters, as it was also confirmed by FE simulations.

Figure 5.3 shows the amplitude of the displacement field in the specimen of the binder 180/200, considering three test runs at a temperature of 20°C, 50°C and 110°C, respectively (the detailed results on these tests will be presented in Section 5.2.4). It is evident from Figure 5.3 how the portion of material affected by the vibration is considerably smaller with increasing temperatures. This is first caused by the fact that at higher temperatures the material becomes softer, the wavelength of the vibration is reduced and less distance is needed to completely damp the waves. In addition, a typically higher viscous behavior is observed for this type of straight run bitumen with increasing temperature, further reducing the distance the waves can travel. In Figure 5.3, the outcomes of the measurements in terms of $|G^*|$ and $\delta$ are reported for completeness, anticipating the results presented later in Section 5.2.4.

![Figure 5.3](image)

**Figure 5.3:** Different shear waves radiation patterns calculated with the FE model at temperatures of 20°C, 50°C, and 110°C, respectively. Colors represent the magnitude of the displacement field. The displacement is normalized with respect to the displacement at the tip of the resonator.
### 5.2.4 General binder characterization

The HFTR can measure the viscoelastic properties of bitumen at approximately 5 kHz, corresponding to the first torsional eigenfrequency of this device \( f^I = 5090 \) Hz in air. A first series of tests has been carried out at different temperatures. The purpose of these tests was to characterize the virgin properties of the binder, before applying any aging procedure. The HFTR was placed into an oven (TSW 120 ED, Salvis AG, Switzerland) together with the specimen, with the lower extremity of the resonator in contact with the specimen. The measurements were acquired as soon as the desired temperatures (20°C to 110°C) were reached by the specimen. Data recording was restricted to the first two hours, in order to minimize the effect of aging that might occur at higher temperatures even for short time periods.

The results of these tests are depicted in Figure 5.4, reporting the values of \( |G^*| \) and \( \delta \) for the temperature range 20°C-110°C. The bitumen undergoes the typical transition from a mainly elastic, at low temperatures, to a mainly viscous behavior that is reached already at 60°C, as can be seen from the value of the phase angle \( \delta \) that approaches 90 degrees. The material reduces its shear modulus considerably with increasing temperatures as it can be seen from the behavior of \( |G^*| \).

This behavior was also highlighted by tests already carried out on straight run bitumens at lower frequencies with standard rheometers\[52, 48, 97\]. The results obtained at high frequencies with the HFTR technique are compared to the results obtained with DMA tests performed with the DSR-50 (a standard rheometer, see Figure 5.4: Material parameters \( |G^*| \) and \( \delta \) evaluated at 5 kHz with HFTR for the bitumen 180/200. Experiments were carried out on virgin samples controlling the temperature of the tests.

![Material parameters |G^*| and δ evaluated at 5 kHz with HFTR for the bitumen 180/200. Experiments were carried out on virgin samples controlling the temperature of the tests.](image-url)
5.2. Laboratory aging tests of bituminous binders

Figure 5.5: Magnitude $|G^*|$ of the complex shear modulus from the HFTR (high frequencies, 5 kHz, points at the right) and the DSR-50 (low frequencies, 0.1-20 Hz, points at the left) for bitumen 180/200. Dashed lines are guidelines for the eye.

Figure 5.6: Phase angle $\delta$ of the complex shear modulus from the HFTR (high frequencies, 5 kHz, points at the right) and the DSR-50 (low frequencies, 0.1-20 Hz, points at the left) for bitumen 180/200. Dashed lines are guidelines for the eye.
Section 5.2.1). The material was characterized with the DSR-50 for the low frequency range (0.1-20Hz), varying the temperature from 20°C to 70°C and controlling the strain amplitude to be 1%. The results obtained with the HFTR at 5 kHz are in line with the low frequency results, in terms of order of magnitude and trend, as shown in Figure 5.5 and Figure 5.6. A continuous increase of the shear modulus $|G^*|$ with frequency is observed, as well as a decrease in phase angle $\delta$ for experiments with 50°C.

5.2.5 Effect of pressure and temperature

The HFTR can be used in accelerated aging tests to quantify the effect of prolonged exposure of specimens to high pressure and temperature. The purpose of this series of tests was to quantify separately the effect of pressure and temperature in the accelerated aging procedures. To this end, a Pressure Aging Vessel (PAV, ATS 504 9-95, Applied Test Systems Inc., USA) was used to simulate aging in the material, controlling pressure and temperature during the tests. As specified in Section 5.2.3, 18 specimens of the bitumen 180/200 were prepared in glass containers and aged in the PAV following three different control profiles identified by the letters (a), (b) and (c).

In the procedure (a), the temperature inside the PAV was increased to 110°C and the pressure was kept to the atmospheric level of 0.1 MPa. In (b) the temperature was left at ambient conditions of 20°C, increasing the pressure inside PAV to 2.1 MPa. Finally, in (c) both temperature and pressure were increased to 110°C and 2.1 MPa, respectively. Each of the procedures (a), (b) and (c) has been applied to 6 specimens. The aging procedures were interrupted after 1 h, 10 h and 20 h extracting 2 specimens for each testing time to monitor the evolution of the material properties during the duration of the test. Tests were conducted once the temperature of the specimens stabilized at 20°C.

Prior to the aging procedures, all the specimens were tested at 20°C in order to obtain reference values, indicated by $|G_0^*| = 44 \pm 0.46$ MPa and $\delta_0 = 18.4 \pm 0.7$ degrees (statistics obtained on a total of 50 measurements on all samples considered). After the conclusion of the test (a), (b), or (c), each specimen was allowed to cool down at ambient temperature and measured again with the HFTR at 20°C.

![Figure 5.7: Samples before and after testing.](image-url)
Figure 5.7 shows the specimens before testing (0), after a temperature controlled test (a), after a pressure controlled test (b) and after a combined pressure and temperature test (c). In Figure 5.7c, the combined effect of pressure and temperature on the specimen is evident, resulting into the formation of air bubbles inside the material and consequently into a volume increase. For the case (c) only, a final 10 minutes of post-heating phase at 110°C and atmospheric pressure was necessary to remove the air inclusions from the material, which would affect the reliability of the HFTR measurements. After the post-heating phase the surface of the specimens was similar to case (b), free from air inclusions.

In Figure 5.8 and Figure 5.9 the results are presented in terms of magnitude and phase angle of the shear modulus, normalized with respect to the corresponding reference values. Each specimen was measured in several locations on the surface exposed to air. The mean value and the standard deviation for each case (a), (b) and (c) are reported in Figure 9 and Figure 10. The standard deviation is caused by the variability of the measurements taken at 10 locations on the specimens (local variability), as well as by the uncertainty related to the HFTR measurement method, as discussed in [71].

The application of a 20 h test on the samples with high pressure only (b) did not produce any significant change in the material properties. The temperature cycle (a) induced an average increase of 4.9% in the shear modulus $|G^*|$ (material hardening) and a decrease in the phase angle $\delta$ of about 9%. A similar behavior was observed applying pressure and temperature at the same time (c). No statistically significant difference was observed between (a) and (c).

The bulk properties of all samples were tested after the aging procedures by removing the top surface layer of material with a heated knife (approximately 5 mm of materials were removed from the surface). The corresponding results are reported in Figure 5.8 and Figure 5.9, on the right. No significant difference can be observed among the samples after the three different treatments (a), (b) and (c) at a depth of approximately 5 mm. Moreover, the values obtained were close to the original reference values $|G_0^*|$ and $\delta_0$. These results suggest that the changes in the material properties take place only in the top surface layer of the specimen, influenced locally by the oxidation process. These results and the questions related to the layered nature of material modifications in the PAV process promoted the investigations presented in the following section.
5. Characterization of Synthetic Materials

**Figure 5.8:** Variation of the shear modulus $|G^*|$ during a 20 h test in the PAV using different aging procedures (a), (b), (c). Standard deviation bars indicated in the figure.

**Figure 5.9:** Variation of the phase angle $\delta$ during a 20 h test aging test in the PAV using different aging procedures (a), (b), (c).
5.2.6 Depth dependence of the material properties

The HFTR can be used to shed some light on the variation of the material properties as a function of the depth of binders exposed to aging procedures. Another glass container (diameter 80 mm, height 150 mm) of 180/200 was placed into an oven, controlling the temperature at 110°C. At this temperature, the bitumen behaves as a perfect viscous material, the phase angle $\delta$ being very close to 90° (see Figure 5). Under these circumstances, the HFTR can be easily lowered into the binder, controlling its vertical position with respect to the free surface (penetration depth $h$) through a travel stage, as indicated in Figure 5.10.

Two thermometers checked the temperatures of the chamber and of the HFTR to avoid any difference which would affect the measurements. The HFTR was lowered into the glass container at different depths and the measurements were acquired after temperature stabilization. Measurements were taken within one hour to minimize the effect of aging observed in the previous section. The variation of resonance frequency of the device with respect to the initial reference configuration $\Delta f_{res}$ and damping characteristic $df$ are reported as a function of the penetration depth $h$ in Figure 5.12. Uncertainty on the depth $h$ of ±0.5 mm, due to adhesion at the sides of the resonator and positioning error of the travel stage, is reported in the plot. The volume of the bitumen was considered to be constant at the pressure $p=1$ bar: no steady increase in the volume caused by air absorbed into the bitumen was observed.
### 5. Characterization of Synthetic Materials

**Figure 5.11:** Validation of the FE model used to interpret the results of the depth-dependence experiment with silicone oil, (Brookfield, USA, calibration oil with homogenous viscosity $\mu=100$ mPas at $20^\circ$, corresponding to $|G^*|=3204$ Pa).

These results can be interpreted in terms of the material parameters using the finite element approach presented in Chapter 3, considering the sensor having lateral contact with the bitumen for a depth $h$. The FE approach returns the expected values of $\Delta f_{res}$ and $df$ for given values of the material parameter $|G^*|$. The whole procedure was validated with a similar experiment with silicone oil (Brookfield, USA, calibration oil with homogenous viscosity $\mu=100$ mPas at $20^\circ$, with no influence of the depth), providing a validation of the proposed approach, as shown in Figure 5.11. The viscosity $\mu$ can be related to the shear modulus $G^*$ at a given angular frequency $\omega = 2\pi f$ by Equation 5.2:

\[
\begin{align*}
G_2 &= \omega \mu & G_1 &= 0 \\
G^* &= j\omega \mu & |G^*| &= \omega \mu
\end{align*}
\]

Considering now the experiment with the bitumen 180/200 at $110^\circ$C, and assuming a constant value of $|G^*|$ for the whole sample, the measured values of $\Delta f_{res}$ and $df$ cannot be fitted with the FE simulation, as shown in Figure 5.12. While a high value of $|G^*|=100000$ Pa fits very well the data up to $h=1$ mm, lower values are needed to fit the measurements at higher depths $h$ (see curve for $|G^*|=25000$ Pa).

These results suggest that a stratification process takes place in the material right after the exposure to high temperatures. The material presents properties that are strongly dependent on the depth. The top layer directly exposed to the
The continuous variation of the material properties as a function of the depth $h$ can be as a first attempt approximated with a discrete distribution, defining different material layers in the FE model. Assuming a step distribution for varying between 100000 Pa and 25000 Pa (as indicated in Figure 5.12 on the right), the experimental results can be rationalized (in Figure 5.12, the experimental data agree to a great extent with the FE results assuming this step distribution). Four different layers were used in this simulation, indicated by I ($0 < h < 1$ mm, $|G^*| = 100000$ Pa), II ($1 < h < 2$ mm, $|G^*| = 60000$ Pa), III ($2 < h < 4$ mm, $|G^*| = 40000$ Pa), IV ($h > 4$ mm, $|G^*| = 25000$ Pa).

Plots from the FE simulations are shown in Figure 5.13 for two different values of the penetration depth $h$, indicating the four different layers I, II, III and IV. As it can be noticed from the amplitude of the displacement field, the top surface layers are characterized by wider shear waves radiation patterns (longer wavelength, longer wave propagation distance) than the internal regions.

This stratification phenomenon takes place in less than 1 h of exposure of the specimens to high temperature. A possible explanation can be found in the diffusion kinetic of oxygen in the material, very fast at the surface, leading to a rapid change in the mechanical properties of the top layers. By continuing this test in the oven for longer time, up to 20 h at 110°C, a progressive material hardening can be observed, as confirmed by the results presented in Poulikakos et al. [71]. Those results showed an increase in the shear modulus during the 20 h in the range of few percents (6%). After the initial stratification phase, the aging process seems to progress slowly. Further discussion on this topic is presented in the next section.
5. Characterization of Synthetic Materials

5.2.7 Discussion

The results obtained with the HFTR on the straight run bituminous binder 180/200 confirmed similar findings obtained with standard shear rheometers. The aging process modifies the mechanical properties of the bitumen, increasing its elastic component, mainly attributed to oxidative aging [48, 49, 52, 76, 97]. The oxidation process increases the content of functional groups and the molecular weight. An increasing content of oxygen-containing functional groups increases bitumen polarity and molecular association, leading to an increase in the stiffness properties of the binders [49].

The results of the aging tests in the PAV have shown that the temperature has a predominant effect in the aging process, as observed applying the procedure (a). The combined application of pressure and temperature was expected to speed up the process, contributing to a faster diffusion of oxygen in the specimen. The results obtained in case (c) contradicted this expectation, being the variation of the material properties and very similar to the one observed in (a). The HFTR could not sense any statistically significant difference between (a) and (c), as the standard deviations of the measurements are comparatively too large to allow a distinction.

A stratification process of the material properties of the binders exposed to high temperatures in the oven was observed. A difference by a factor of 4 in the shear modulus was estimated by the FE calculations between the top surface layer and the bulk regions at 110°C. The tests executed at 20°C on aged samples reported in Section 5.2.5 had shown a smaller difference between a measurement on the top and a measurement taken after removal of the first 5 mm of material. After cooling down the samples at 20°C, the difference observed for |G*| was only 4.9%. This suggests
that the oxidation process influences the mechanical properties in a different way depending on the temperature. At high temperature, where the bitumen behaves like a fluid, the oxidation leads to a stratification of the material properties more pronounced than at low temperature, where the bitumen behaves like a solid. An extrapolation of the dependence of the shear modulus on the depth from high to low temperatures is not possible.

The presence of the stratification has also important consequences on the results presented in Figure 5.4, where the bitumen was characterized in the temperature range 20°-110°C. The results presented correspond to measurements executed on the surface of the specimens, mainly representing the properties of the top layers. As seen at 110°C in the depth dependence characterization of the material properties, the shear modulus obtained measuring the top layer will be higher than the one obtained measuring the internal regions (varying from |$G^\ast$| = 100 000 Pa on the surface to |$G^\ast$| = 25 000 Pa in the internal regions). A variability of the material properties with the depth must be expected for all the tests executed. The internal regions of the bitumen will be characterized by smaller values of |$G^\ast$| than its surface, and this difference will assume more importance with increasing temperature.

Further investigation is needed to shed some light on this phenomenon. Due to the impossibility to penetrate into the bitumen samples at low temperature (where the material behaves like a solid), and the difficulties encountered in removing uniformly the superficial layers from the binder samples, other experimental set-up will be required.

### 5.3 The Long term aging of bituminous binders

The possibility to monitor aging of pavements and bituminous binders directly on the operative field, under their typical environmental and service conditions, can contribute to better understand the factors influencing the aging process, can be used for material optimization and for maintenance planning. However, this is not an easy task due to the experimental problems that arise in measuring the rheological properties directly on the field. In fact, most common rheometers cannot be employed. The HFTR is a useful device to study the time evolution of the rheological properties of bituminous binders during field tests. In comparison with prevailing rheometers, the HFTR is inexpensive and, due to its portability and operative frequency (5 kHz) that allows measurements to be unaffected by traffic induced low frequencies, is a useful tool for continuous monitoring in field tests.

In this section, the results obtained from a series of measurements with the HFTR for field tests on an asphaltic plug joint located on a highway bridge are analyzed, monitoring the changes in the viscoelastic behavior of the binder (an elastomeric SBS, styrene-butadiene-styrene [13], polymer modified bitumen) over three years of field tests. The experimental results of these aging tests were already presented by Poulikakos et al. [70] in terms of changes in equivalent stiffness and damping coefficients. Here, they are interpreted quantitatively in terms of the variation in
the material complex shear modulus $G^*$ as a function of temperature and time of exposure, giving therefore useful insight into the rheological properties of polymer modified bitumen during the aging process.

5.3.1 Plug joint under investigation

Asphaltic plug joints are widely used for accommodating structural movement in highway bridges. The primary advantage of this system with respect to other type of joints (finger joints or butt joints) is ease of installation and repair. Other advantages include the low cost of installation and repair, low instance of snowplow damage, and that it can be cold milled.

The asphaltic plug joint under investigation is located on a highway bridge close to Basel, Switzerland, and was constructed in 1998. The plug joint consists of binder and stones of small aggregate size that form a load bearing skeleton (see Figure 5.14). The construction of this plug joint was done in four layers. The lower layers one to three use 20 mm aggregate size whereas the last layer has 14 mm aggregates. The surface of the top layer that is trafficked is covered with 3-5 mm aggregates. The air void content is extremely low, close to zero. The binder is an

![Figure 5.14: Schematic of the asphaltic plug joint under investigation.](image)
elastomeric polymer modified bitumen (PMB), produced from B40/50 containing 12% of styrene-butadiene-styrene (SBS), with density $\rho = 1039 \text{ kg/m}^3$. This is about the double of the amount of SBS normally used for pavement binders. The binder was poured in layers of 30-40 mm at a temperature of 180°C with an equal layer of hot stones (max 22 mm aggregate size) added immediately. Then, this layer was allowed to cool to about 80°C and another layer was added to a total thickness of 16 cm. Once the HFTR was placed on the surface of the asphaltic plug joint and was free to move vertically, it began to sink slowly due to its own weight. The vertical downward displacement reached a maximum value of approximately $h = 4$ mm that remains stable as it was also confirmed by prior laboratory experiments [70].

5.3.2 The long-term field test

The long-term field test started in 1998 on the highway plug joint described in Section 5.3.1. The measurements were taken continuously for three years (1998 to 2000), recording also the temperature changes in order to monitor the temperature-dependent behavior of the binder.

The temperature indicated in this study represents the reading of a temperature sensor located inside the HFTR, 10 cm above the surface of the plug joint. Thermal equilibrium was assumed in this analysis between the HFTR and the plug joint. For each year, the results recorded during July, August and September, are compared, presenting temperatures ranging from 10°C to almost 50°C. In these months, the higher temperature observed (with respect to cold seasons) allows a wider temperature characterization of the plug joint material. Furthermore, material aging and performance deterioration occurs at temperatures higher than 30°C [49].

Figure 5.15 and Figure 5.16 show the values of the measured quantities ($f_{res}$, resonance frequency, and $df$, damping characteristics) during the tests for the three years, as a function of the recorded temperature. A regression analysis of the recorded data is presented in Figure 5.17 and Figure 5.18; the curves represent the mean value of the measurements obtained at the corresponding temperatures, the corresponding standard deviation is reported beside each curve ($\bar{\sigma}$ represents the standard deviation in percent with respect to the mean value).

From the measurements of $f_{res}$ and $df$, using a mechanical model of the resonator (described in Appendix A.2), the value of the torsional mechanical impedance can be extracted. Figure 5.19 and Figure 5.20 show the torsional mechanical impedance, magnitude $|Z_T|$ and phase , obtained from the recorded data.
5. Characterization of Synthetic Materials

**Figure 5.15:** Resonance frequency $f_{\text{res}}$ as a function of the temperature recorded during three years.

**Figure 5.16:** Damping characteristics $d_f$ as a function of the temperature recorded during three years.

**Figure 5.17:** Regression curves of the resonance frequency $f_{\text{res}}$ and damping characteristics $d_f$ as a function of temperature recorded during three years. Standard deviation $\sigma$ in percentage of the mean value reported beside each curve.

**Figure 5.18:** Regression curves of the resonance frequency $f_{\text{res}}$ and damping characteristics $d_f$ as a function of temperature recorded during three years. Standard deviation $\sigma$ in percentage of the mean value reported beside each curve.
5.3.3 Extraction of the material parameters with the FE model

The rheological properties of the plug joint can be extracted in terms of the complex shear modulus $|G^*|$ from the measured values of $Z_T$, using the FE model described in Section 3.3.1. The FE model includes both the HFTR sensor, modeled in detail in each component, and the material of the plug joint, as shown in Figure 3.10. The contact takes place on the lower end of the vibrator and on the lateral surface for a total height $h = 4$ mm. The plug joint was modeled as a homogenous material, assuming the stones to be uniformly distributed into the binder (density $\rho = 2367$ kg/m$^3$, $\nu = 0.35$). This assumption allows obtaining an average value of $G^*$ representing the rheological behavior of the whole plug joint.

The geometric and material data used for the FE model of the HFTR for field tests have been reported in Figure 2.13 and Table 2.2. The changes in temperature during the tests that also influence the dynamic behavior of the sensor (with and without contact with the medium) are also considered. The storage shear modulus $|G^*_s|$ of the HFTR sensor is considered to be temperature dependent (see Table 2.2), compensating the variation of $f_{res}$ and $df$ due to temperature changes in the sensor alone.

In the FE simulations, the complex shear modulus of the plug joint material $G^*$ is searched for, in order to match the experimental results reported in Section 5.3.2. The results of the optimization are reported in Figure 5.21 and Figure 5.22, where the values of $|G^*|$ and of the phase angle $\delta$ evaluated using the FE model are plotted as a function of the temperature. Figure 5.21 shows a progressive increase during the three years of the magnitude of shear modulus $|G^*|$ in the plug joint. The increase in the modulus occurs at any temperature, with a relative change larger at higher temperatures. Figure 5.22 shows the evolution during the test of the phase angle $\delta$.
that quantifies the amount of viscous and elastic component in $G^*$. At the beginning of the test the phase angle decreases at high temperatures, going from 27 degrees at 10°C to 22 degrees at 50°C. After exposure to atmospheric conditions this angle shows a strong increment, especially at higher temperatures, reaching a final value of 54 degrees at 50°C in year 2000, which is more than double of the initial value of 22 degrees in 1998. The behavior reported at the beginning of the test, in 1998, is often observed in SBS polymer modified binders: the phase angle decreases with increasing temperatures\cite{49, 2}. The addition of the elastomeric polymer into the base bitumen significantly improves the elastic response of the binder. The increase in the elastic response, especially at high temperatures, can be attributed to the viscosity of the base bitumen being low enough to allow the elastic network of the polymer to influence strongly the mechanical properties of the binder\cite{25, 21}. The prolonged exposure to environmental conditions is responsible in the next two years for a material aging that leads to an increase of the phase angle $\delta$, manifesting itself with a higher viscous behavior at high temperatures than the one showed at the beginning of the test.

Considering the case of $T = 20^\circ$C in year 2000, the vibration pattern generated by the HFTR in the plug joint is depicted in Figure 5.23. The FE simulation was run specifying a value of $|G^*| = 316$ MPa and $\delta = 34$ degrees, resulting into the corresponding measurement values of $f_{res} = 5420$ Hz and $df = 98$ Hz recorded in the experiment in year 2000 at 20° (see Figure 5.17 and Figure 5.18). From the magnitude plotted in Figure 5.23, a pronounced decay of the shear waves excited in the material is evident: the forced torsional oscillations do not penetrate in the plug joint material more than twice the radius of the vibrator, due to the high damping present in the binder.
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Figure 5.23: Normalized displacement field $|u_\phi|$ of the system HFTR sensor + material at 20°C in year 2000, evaluated from the FE simulation, corresponding to values of $|G^*| = 316$ MPa and $\delta = 34$ degrees. Values extracted from Figure 5.21 and Figure 5.22.

5.3.4 Discussion

All the results obtained in this study with the HFTR represent rheological measurements at a frequency of about 5400 Hz (determined by the second torsional eigenfrequency of the sensor used in this analysis). The values of the complex shear modulus $G^*$ obtained at high frequency are higher than those obtained with conventional rheometers for DMA analysis operating in the range 0.1-100 Hz. According to the frequency-temperature superposition principle [21], any increase in the frequency corresponds to a temperature decrease in the test. Measurements at other frequencies can only be obtained by changing the torsional resonance of the HFTR sensor, i.e. changing its design.

The material hardening observed during the three years with a progressive increase in the complex shear modulus $G^*$ is in line with results obtained in laboratory aging tests on similar SBS polymer modified bitumen using standard procedures (as the thin film oven test, TFOT, rolled thin film oven test, RTFOT, and the modified rolled thin film oven test, MRFTOT [2, 48, 49, 76]). The desired effect of polymer modification is to provide a polymer network that imparts elastic stability at higher temperatures, indicated by a decrease of the phase angle $\delta$, observed at the beginning of the test in 1998. This feature contributes to improve the resistance to
permanent deformation of the pavement in the form of ruts or corrugations, typical observed in straight run bitumen (without polymer modification) due to the higher viscous behavior at high temperature.

The rheological changes observed during the field test may be explained by polymer degradation in the binder. Breakdown of SBS reduces the density of the polymer network, and consequently, the effectiveness of the polymer to modify the bitumen rheology is reduced, showing a higher viscous behavior typical of a base bitumen [48, 49]. The addition of PMB to a base bitumen is usually made to reduce the temperature susceptibility at least in a certain temperature (or frequency) range [48, 76]. Our tests showed that this effect is strongly reduced after 3 years of exposure to environmental conditions. The rheological behavior changes from elastic to viscous depending on the temperature in a much more pronounced way than at the beginning of the test.

A source of uncertainties in the measurements can be attributed to the temperature registered by the sensor placed inside the HFTR during the experiments. Although thermal equilibrium was assumed between HFTR and plug joint, small temperature differences might exist, leading to a correction in the temperature scale of the experimental results presented in this study. The assumption of homogenous material was made to model the interaction with the plug joint, considering its material properties to be uniformly distributed. The effect of concentrated stiffness brought by the stones cannot be modeled without knowing their location. Further studies with the FE approach could consider the distribution of the stones in the plug joint, using CT-scans images on a cross section, to quantify the influence of inhomogeneity on the measurements with HFTR.

5.4 Electroactive Polymers

In this section, the dynamic torsion test technique is applied to the characterization of electroactive polymers (EAPs). Collaboration with Michael Wissler, EMPA, was started in 2005, aiming at the mechanical characterization over a wide frequency range of specimens of a specific EAP, a dielectric elastomer. In this joint project, combined tests on the same dielectric elastomer were carried out: (i) testing the material with the dynamic torsion test and (ii) performing uniaxial tensile tests with standard machines. Tests were carried out on EAP specimens at different levels of pre-strain (which reached up 500% of nominal strain) and at different frequencies (quasi-static uniaxial relaxation tests for the low frequencies, 0.01-1 Hz, and dynamic torsion tests for the range 1-12 kHz).

The specimens of EAPs considered in these tests consisted of thin membranes of different thickness \( q \), ranging from 1000 \( \mu m \) to 40 \( \mu m \). The capability of the dynamic torsion test in determining the mechanical properties of membrane-like specimens is demonstrated in this section. Both TRD and HFTR were employed in this investigation.
5.4. Electroactive Polymers

Figure 5.24: Specimen of dielectric polymer VHB 4910 fixed to a circular frame (internal radius 75 mm). Specimens with different thickness can be obtained by following the procedure outlined by Zhang et al. [99], applying different levels of pre-strain.

5.4.1 Samples characteristics and preparation

The dielectric elastomer considered in the present work is a commercial available acrylic elastomer VHB 4910 (3M, USA). This material has the capability of undergoing large strains (up to 500% nominal strain) and is characterized by a high elastic energy density [7]. The VHB 4910 has a density $\rho = 960$ kg/m$^3$ and exhibits a nearly incompressible material behavior, confirmed by the tests, thus is $\nu \approx 0.5$. The material is obtained in 1 mm thick foils from the supplier. Specimens of different thickness can be obtained by pre-stretching the 1 mm thick foil radially, according to a procedure outlined by Zhang et al. [99], and constrain them to a plastic circular frame, as shown in Figure 5.24. Following this procedure, thin film-specimens of different thickness $q$ can be obtained, each thickness corresponding to a different level of pre-strain. The elastomer film can be fixed to a plastic circular frame (radius 75 mm), and tested with the dynamic torsion test in correspondence to its center (see black marker in Figure 5.24).

In Table 5.1, the characteristics of the specimens considered in this analysis are reported. Specimen $\sharp 0$ corresponds to the initial undeformed foil obtained from the supplier ($q=1000$ µm), with a stretch ratio $\lambda=1$ (the stretch ratio $\lambda$ is defined as the current length divided by the original length in the principal direction of deformation, see Appendix B.1). Specimens $\sharp A$, B, and C were obtained by stretching the original foil radially up to a stretch ratio $\lambda=3$, 4 and 5, respectively. The relation between the pre-strain applied to the film and the thickness $q$ after stretching, in case
Table 5.1: Characteristics of the specimens of dielectric elastomer VHB 4910.

<table>
<thead>
<tr>
<th>( \sharp )</th>
<th>Sample type</th>
<th>( \lambda ) [-]</th>
<th>( q ) [( \mu \text{m} )]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Undeformed film</td>
<td>1</td>
<td>1000</td>
</tr>
<tr>
<td>A</td>
<td>300% pre-strained film</td>
<td>3</td>
<td>111</td>
</tr>
<tr>
<td>B</td>
<td>400% pre-strained film</td>
<td>4</td>
<td>63</td>
</tr>
<tr>
<td>C</td>
<td>500% pre-strained film</td>
<td>5</td>
<td>40</td>
</tr>
</tbody>
</table>

of uniform radial deformation, can be found by imposing an isochoric deformation as:

\[
q(\lambda) = \frac{1}{\lambda^2} \cdot q(\lambda = 1).
\] (5.3)

The thickness of the probes was checked after their preparation using a thickness sensor (Mitutoyo ID-F125/150).

5.4.2 Characterization of EAPs with dynamic torsion tests

The configuration of the tests executed on thin membrane-like specimens of the dielectric elastomer analyzed is reported in Figure 5.25. The torsional vibrating sensor is placed on the thin membrane and exerts torsional oscillations in correspondence to its eigenfrequency \( f \). The particular EAP considered in this analysis is used by 3M as an adhesive tape and for vibration isolation, therefore it adheres very easily to steel materials. Good adherence between the tip of the resonator and the material was therefore observed for both TRD and HFTR sensors. For the TRD sensor, vacuum clamping was in this case not necessary to ensure the non-slip condition.

The EAP film is modeled as a homogenous, linear viscoelastic material with a constant thickness \( q \), clamped at the external boundaries to the frame. The finite element approach described in Chapter 3 can be used to describe the interaction between the vibrating sensor and the EAP film. Due to the finite radial dimensions of the specimens, it is important to check on possible stationary effects resulting from wave reflections in correspondence to the frame. In Figure 5.25, the FE simulation of the interaction is reported, with colors indicating the magnitude of the induced displacement field. Due to the high damping properties observed in this dielectric elastomer (with \( \delta \approx 0.6 \), anticipating the results of the tests), the radial dimensions of the specimens are large enough to prevent any reflection at the boundaries, as it was also confirmed by measurements of the displacement field with laser interferometry. Since stationary effects were excluded with FE calculations, the analytical model developed for thin membranes in Section 3.2.2 can be used to describe this interaction, with advantages in terms of computational time with respect to FE. Assuming a constant thickness of the membrane \( q \), the mechanical parameters \( G^* \) and \( \delta \) are related to values of \( df \) and \( \Delta f_{res} \) obtained from the tests.
Figure 5.25: FE model of the dynamic torsion test executed on EAPs membrane specimens. The thin membranes of thickness $q$ is fixed at its extremity to a circular ring. Colors indicate the normalized vibration amplitude in the specimen.

**Frequency characterization**

Specimen $\#0$ (undeformed film of thickness $q = 1000 \, \mu m$) was tested using both TRD and HFTR sensors. The results of these tests are reported in Figures 5.26 and 5.27, showing the values obtained in the high frequency domain with the TRD (four testing frequencies considered, $f^{(I)} = 1300$, $f^{(II)} = 6650$, $f^{(IV)} = 9330$, $f^{(V)} = 12150 \, Hz$) and with the HFTR (only one testing frequency, $f^{(II)} = 5090 \, Hz$). The results are presented in terms of $|G^*|$ and $\delta$ in the frequency domain. The standard deviation reported in the plot corresponds to 10 measurements executed on the same specimen in different locations in the central area of the specimen (at least 40 mm far from the frame).

The results obtained with the HFTR are in line with those obtained with the TRD, confirming that the reliability of the measurement technique is independent from the sensor used. An evident increase of the shear modulus $|G^*|$ at high frequencies is shown (a phenomenon observed also in the silicone and polymer rubbers in Section 4.3, while the phase angle seems to remain relatively stable around a value of $0.66 \pm 0.11$.

**Pre-strain dependence**

The different specimens $\#0$, A, B and C are characterized by a different thickness and pre-strain level, as previously reported in Table 5.1. The dynamic torsion test was executed as usual by prescribing a small vibration amplitude (1 $\mu m$ maximum amplitude at the tip), which corresponds to a small perturbation (1.2%) around the
5. Characterization of Synthetic Materials

Figure 5.26: Values of $|G^*|$ obtained from tests on VHB 4910 for specimen $\sharp 0$ of ($q=1000 \ \mu m$, without pre-strain) with TRD and HFTR. Standard deviation referred to 10 measurements executed in different location on the central area of the same specimen.

Figure 5.27: Values of the phase angle $\delta$ obtained from tests on VHB 4910 for specimen $\sharp 0$ of ($q=1000 \ \mu m$, without pre-strain) with TRD and HFTR. Standard deviation referred to 10 measurements executed in different location on the central area of the same specimen.
pre-strain level of the probe considered. The material parameter measured in the
dynamic torsion test is indeed a tangent shear modulus $|G^*|$, related to the
pre-stretch value $\lambda$ applied to the specimens before testing. This value will vary upon
the pre-stretch value prescribed, depending on the nonlinear characteristics of the
material analyzed.

The pre-stretch level has important consequences on the mechanical character-
istics of the EAP, especially at the values considered in this analysis, which vary
from 1 to 5. Results were collected on specimens $\#0, A, B, C$, corresponding to the
stretch level of 1, 3, 4 and 5, respectively. In Figure 5.28, the results are grouped in
five different sets that correspond to the testing frequencies of the TRD and HFTR.
Within each set, the frequency is constant and the value of the pre-stretch varies.
An increase in the tangent shear modulus $|G^*|$ with increasing pre-stretch level is
registered in correspondence to each testing frequency. At 12150 Hz, the scatter
registered in the measurements does not allow identifying a clear increasing trend.

By considering the same pre-stretch level (i.e. within each specimen $\#0$, A, B,
and C), an increasing trend for $|G^*|$ is found for all the stretch levels considered.
For $\lambda$=3, 4 and 5, a frequency dependent behavior similar to what was presented in
Figure 5.26 for $\lambda$ = 1 can be observed.

The phase angle $\delta$ estimated at different pre-stretch levels shows a behavior sim-
ilar to the one reported in Figure 5.27 for $\lambda$ = 1, remaining confined between mean
values 0.66 and 0.75 in the four cases analyzed. Because of the higher scatter regis-
tered in the measurements of the phase angle, differences in the damping properties
of the EAP could not be detected with two devices considered, the sensitivity of the
device being too small to identify clear trends.

5.4.3 Comparison with standard uniaxial tests

A series of standard uniaxial tests on the same dielectric elastomer have been carried
out at the EMPA, by Michael Wissler. Relaxation tests on VHB 4190 specimens
have been executed to determine the hyperelastic and viscoelastic behavior of this
elastomer at high deformation levels (up to 500%). The results of these tests and
the description of the hyperelastic model used for this material can be found in [93].
A brief summary of the results is reported for completeness in Appendix B.3.

From uniaxial tests, a Yeoh hyperelastic model [95] is obtained. The viscoelastic
behavior of the material is described assuming quasi-linear viscoelasticity [22] using
Prony series [85]. From the hyperelastic model of the dielectric elastomer evalu-
ated in [93], the tangent instantaneous shear modulus $G_0(\lambda)$ can be extracted in
correspondence to a given value of the pre-stretch $\lambda$ (see Appendix B.3 for further
details). In Figure 5.29, $G_0$ is reported for the stretch interval $1 < \lambda < 5$. The trend
observed in the instantaneous shear modulus $G_0$, increasing with $\lambda$, is similar to
what has been observed at high frequencies with the dynamic torsion test, reported
in Figure 5.28. However, if the trend is similar, a direct quantitative comparison
between the relative increase found with the dynamic torsion test and the one ex-
pected from the uniaxial tests is not possible. The factor 2.5 found in uniaxial tests
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Figure 5.28: Value of the tangent shear modulus $|G^*_\lambda|$ obtained at different values of the pre-stretch $\lambda$. Results are grouped by the same testing frequency.

Figure 5.29: Instantaneous tangent shear modulus $G_0(\lambda)$ obtained from the Yeoh model of the VHB 4910 [93].
between the linearized shear modulus at $\lambda = 1$ with respect to the value at $\lambda = 5$ cannot be found in the results obtained at high frequency with the dynamic torsion test.

$G_0$ represents the instantaneous elastic response of the dielectric elastomer, and does not include the relaxation process that takes place in time, which is modeled with Prony series. The results obtained from quasi-static experiments can be converted in the frequency domain using the complex notation for $G^*$ by using Equation 5.4:

$$G^*(\omega) = G(0) + \int_0^\infty e^{-j\omega\tau} \dot{G}^{(0)}(\tau) d\tau \quad (5.4)$$

where $G(t)$ is the linearized relaxation shear modulus found in the uniaxial tests, following the same procedure previously used in Section 4.4.5.

In Figure 5.30, the results obtained with the uniaxial tests are plotted in the frequency domain and compared with those obtained from the dynamic torsion tests at four different stretch levels ($\lambda = 1, 3, 4$ and 5). The results obtained from the uniaxial tests can be considered reliable up to a frequency of 1 Hz (due to the type of relaxation test performed), and are reported at the left of the diagram. On the right side of the diagram, the results obtained at high frequency with the dynamic torsion test are reported (dotted lines are guidelines for the eye). From these findings, an exponential dependence of the shear modulus $|G^*|$ with the frequency can be

![Figure 5.30: Comparison of the results obtained with standard uniaxial tests (on the left side) and with dynamic torsion tests (on the right side) at four different levels of stretch ratio $\lambda=1,3,4$ and 5.](image)
5. Characterization of Synthetic Materials

assumed. The phase angle evaluated from the uniaxial relaxation test varies between 0.33 and 0.57 in the low frequency region considered (0.01-1 Hz) (see Appendix B.3).

5.4.4 Discussion

Results obtained with the dynamic torsion test on dielectric elastomer VHB 4910 have shown the frequency dependent behavior of this material. Increase in the magnitude of the shear modulus $|G^*|$ was observed from low frequencies (results obtained with relaxation tests reliable up to 0.1 Hz) to high frequencies (1-12 kHz with TRD and HFTR). The damping behavior of the material was not considerably influenced by the strain rate and by the strain level. The phase angle $\delta$ obtained from uniaxial tests was similar to the one obtained with the dynamic torsion test at high frequencies. A constant $Q$-factor model [22] could in principle be used to reproduce the loss mechanism in the material at different frequencies and strain levels.

The two sensors considered in the analysis of EAPs thin membrane specimens, both TRD and HFTR, has proved to be able to test these viscoelastic materials. Results obtained from the two sensors were consistent, showing the reliability of the technique. A comparison with the results obtained at low frequency from uniaxial tests showed that the results are in line with the expectations, showing a constant increase of the shear modulus at high frequencies.

5.5 Conclusions

The results presented in this chapter have demonstrated the effectiveness of the dynamic torsion test in the mechanical characterization of synthetic materials. This measurement technique was applied in studying the aging phenomena in bituminous binders and in the viscoelastic characterization of electroactive elastomers.

The rheological properties of the straight run bituminous binder 180/200 were characterized in terms of the complex shear modulus at low frequencies and at 5 kHz, for the temperature range 20-110°C. The HFTRr was used to study separately the effects of pressure and temperature in aging the asphalt binder, showing a predominant effect of the temperature in the oxidation process. It was not possible to discern the influence of the combined effect of pressure and temperature from the one with temperature only. The rheological properties of samples exposed to high temperature were characterized as a function of the depth, showing a stratification of the material properties. The top surface layers of the specimen were considerably stiffer than the internal layers not directly exposed to air.

The possibility to monitor directly on field the rheological properties of bituminous binders by means of the HFTR has been demonstrated. The evolution of the mechanical properties of a highway plug joint have been monitored during three years of tests, showing the changes in the rheological properties of the asphalt binder after exposure to environmental conditions. The aging observed over the three years
on a SBS polymer modified bitumen plug joint material manifested itself in material hardening, with an increment of the complex shear modulus $G^*$, and in a progressive increase of the temperature susceptibility of the bitumen mixture, evidenced by a more pronounced viscous component.

The TRD and HFTR were employed in the characterization of thin, membrane-like specimens of a particular electroactive polymer, the dielectric elastomer VHB 4910. The dielectric elastomer samples were characterized at different frequencies (1-12 kHz) and at different levels of pre-strain ($\lambda = 1 - 5$), showing a nonlinear behavior similar to what was expected from comparative quasi-static uniaxial tests.

Results have shown that the dynamic torsion test can be applied to a variety of geometries and materials and serve as a useful tool for a fast and precise determination of the mechanical properties of synthetic materials at high frequencies.
5. Characterization of Synthetic Materials
Conclusions and Outlook

Summary of achievements

A measurement technique based on forced torsional oscillations induced by a vibrating sensor on a viscoelastic medium has been presented in this work. The dynamic torsion test allows a characterization of the linear viscoelastic properties of soft biological and synthetic materials. The rheological behavior is quantified in terms of the complex shear modulus $G^*$ in the frequency range 1-10 kHz.

In Chapter 2, the design of the torsional vibrating sensors used, along with the measurement principle, was introduced. Different sensors have been considered: the TRD for soft biological tissues and the HFTR for bituminous binders. The mechanical models presented reproduce their dynamic behavior delivering results that agreed to a wide extent with the experimental results.

The analytical and finite element approach used to model the interaction between the vibrating sensors and the viscoelastic material samples analyzed have been described in Chapter 3. The analytical solution was used to solve simple contact geometries and as a reference for validation of the numerical, finite element approach. The latter can be used to model also those cases, in which the contact geometry and the size of the material samples do not allow an analytical solution, representing a useful design tool for other similar sensors based on forced torsional oscillations.

In Chapter 4 the results obtained from tests on soft biological tissues ex-vivo were presented. The validation of the experimental technique with silicone phantoms proved its reliability. The results obtained on soft internal organs showed the effectiveness of the technique and contributed to extend the knowledge on the rheology of soft tissues in the high frequency domain. A comparison with results from quasi-static experiments has been proposed for both biological and rubber materials.

The dynamic torsion test, originally developed to measure the viscoelastic properties of soft biological tissues, has shown to be a useful method to study the rheology of soft, synthetic materials, such as silicone and polymer rubbers, bituminous
binders, electroactive polymers. In Chapter 5, the aging phenomenon of bituminous binders was a subject of investigation: the evolution of the mechanical properties of the binders undergoing aging process was quantified.

**Final remarks on the measurement technique**

The experience gained in testing soft biological tissues and synthetic materials lead to important considerations that are summarized here. A series of advantages can be highlighted:

(i) the measurement is fast and local, it requires only few seconds to be executed (60 s for a complete measurement, including the preparation time) and comes in contact only with a small portion of the material sample analyzed (few cm³ or even less, depending on the characteristics of the sample). The capability of the TRD to distinguish inhomogeneities within the same material sample allows to envisage possible clinical application as a diagnostic tool, linking in the future the material properties to different pathologies in the same way as presented by Nava [60] with the Aspiration Experiment on human livers,

(ii) the method proposed permits a reliable and quantitative measurement of the rheological properties in the high frequency domain (1-12 kHz), where other techniques are limited. The TRD represents an alternative non-destructive test for soft biological tissue characterization above the limits of magnetic resonance elastography (600 Hz). Furthermore, it represents a valid alternative to DMA, useful in characterizing soft polymers at frequencies up to 12 kHz with the advantage of simple and fast testing and data analysis,

(iii) in addition to the main application for soft biological tissues, the dynamic torsion test has shown to be efficient in testing synthetic materials such as silicone rubbers, bituminous binders and electroactive polymers. The precision in testing these materials was considerably higher than what was observed for soft tissues. The devices based on this measurement method, due to the easy portability and to the inexpensive construction, can be used as fingerprinting tools for quality control and on-line monitoring of the mechanical properties of soft viscoelastic solids.

The dynamic torsion test proposed in this work has showed promising results and particular features that can make it an interesting tool for soft material characterization. However, a series of problems and disadvantages, typical of this measurement technique, must be kept in mind:

(i) the control electronics and measurement principle was entirely based on the know-how previously developed at the Institute of Mechanical Systems, ETH Zurich, by Sayir et al. [79]. This method was proved to be very efficient in measuring viscosity of fluid and suspensions, but has intrinsic problems in
determining combined measurements of elasticity and viscosity in solids, due to the high precision required for the measurement of $\Delta f_{res}$ and its susceptibility to temperature variations, that strongly affect the outcome of the results in the inverse material characterization, as discussed in Section 4.5. Furthermore, this measurement method is not spectroscopic: only a discrete measurement of the mechanical properties in correspondence to the torsional eigenfrequencies of the sensors used can be obtained. Further development of this technique for soft tissue testing should increase the number of testing frequencies and reduce the lower frequency limit, that is of particular interest in modeling the mechanical response of soft tissues to physiological loads,

(ii) the local characteristic of the measurement method proposed can also be considered a limit, rather than an advantage, in some cases. Since the penetration depth of the shear waves does not exceed 1-2 cm, this can represent a disadvantage if one aims at measuring the mechanical properties in the bulk region of soft internal organs,

(iii) the precision of the measurement is limited by the accuracy of the control electronics and the characteristics of the sensors used. Testing the mechanical properties of viscoelastic materials with $|G''| \approx 3$-100 kPa in the range 1-12 kHz is a challenging task. The reproduction in the experiments of the boundary conditions assumed in the mechanical model represents also a problem and a source of uncertainties. These uncertainties will increase for in vivo tests in the operation room, due to the different variables that could affect the test (e.g. the motion of the surgeon or the cardiac pulse of the patient, temperature difference between sensor and soft tissues) and must be considered in future developments.

**Outlook: Micro-TRD for laparoscopic surgery**

The application of the TRD in clinical studies on soft biological tissues requires the development of a sensor of reduced dimensions that can easily be handled by a surgeon in the operation room during in vivo tests. This requirement has lead to the development of a new, miniaturized device, the micro-TRD, suitable to be used during laparoscopic surgery.

The prototype of this device, reported in Figure 6.1, was realized at the Institute of Mechanical Systems, ETH Zurich, by Dr. Klaus Häusler. The device consists of a torsional resonator of reduced dimensions (steel tube of outer diameter 500 $\mu$m and inner diameter 300 $\mu$m), protected by an external case (diameter 9 mm) that can be inserted into a 10-mm trocar and used during laparoscopic surgery. Figure 6.2 shows the complete device inserted into a 10-mm trocar case. On the left side of Figure 6.2, the construction scheme of this miniaturized device is presented. The pressure inside the inner part of the steel tube resonator can be controlled, in order to achieve adherence with the material. In this case, instead of a disc with micro-
openings at the tip of the resonator, the contact lays on a circular crown of outer diameter 500 µm and inner diameter 300 µm.

One of the main challenges that have to be solved for this device is the low sensitivity (due to the reduced size of the contact area) and the difficulty to verify the boundary condition of adherence. Furthermore, pure torsional motion at the tip was difficult to be observed (a bending component was also measured during preliminary tests), due to the difficulty of manufacturing a balanced resonator of dimension in the order of few millimeters. This affects the impedance measurement and the inverse material characterization is compromised. Further research must be carried out to improve the design of the device and overcome the experimental problems outlined above.
Appendix A

Modeling the mechanical behavior of the sensors

A.1 Analytical model of the TRD

The Torsional Resonator Device can be modeled as a continuous deformable structure to accurately predict its dynamic behavior. In this approach, the tube is considered as a continuous beam, characterized by the shear modulus $G_s^*$, the density $\rho_s$, and the polar moment of inertia $J_p$. The magnets bonded to the tube and the contact disc are considered as discrete elements, using lumped inertia moments $J_m$ and $J_d$ to represent them. The contact with a viscoelastic medium is modeled with the introduction of a torsional impedance $Z_T$ in correspondence with the contact disc. Figure A.1 represents a mechanical scheme of the sensor. The excitation torque applied by the electromagnetic transducer $M_e = M_e e^{j\omega t}$ drives the system in torsional vibrations with the angular frequency $\omega = 2\pi f$.

$$M_e = Z_T \pi a^2 \hat{\theta}_3 (L_3, t)$$

![Mechanical scheme of the TRD interacting with a medium with torsional impedance $Z_T$.](image)

Figure A.1: Mechanical scheme of the TRD interacting with a medium with torsional impedance $Z_T$.  

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The behavior of the tube, modeled as a continuous beam, is described by the classical wave equation in one dimension. In case of harmonic time dependence with angular frequency $\omega$, it follows that:

$$\theta_{,xx} + k_s^2 \ddot{\theta} = 0$$  \hspace{1cm} (A.1)$$

$$k_s^2 = \frac{\omega^2}{c_s^2} = \frac{\omega^2}{G_s \rho_s}$$  \hspace{1cm} (A.2)$$

where $c_s$ is the shear wave speed of the material and $k_s$ is the wave number in the tube. The viscoelasticity in the tube is taken into account considering the loss factor $\eta_s$ of the material, as described in Equation A.3:

$$G_s^* = G_{1s} + j G_{2s} = G_{1s} \left(1 + j \frac{G_{2s}}{G_{1s}}\right)$$  \hspace{1cm} (A.3)$$

$$G_s^* = G_{1s}(1 + j \eta_s) \quad \text{with} \quad \eta_s = \frac{1}{Q_s}$$  \hspace{1cm} (A.4)$$

The value of $\eta_s$ is usually very small in metals, and is linked to the material quality factor $Q_s$ by Equation A.4. In the current design, the brass alloy chosen for the TRD has a very high $Q_s \approx 20000$. Other typical values of $Q_s$ can be found in the literature [35] or directly after performing experiments on the material.

The material damping in the tube is considered by specifying the value of $G_s^*$. All the other intrinsic damping sources (that will be further discussed) are grouped in a lumped damping element $d_s$ placed in the position of the excitation coil (see Figure A.1). The value of $d_s$ must be extracted from the experiments in a calibration run, and varies depending on the mode excited.

![Figure A.2: Balance of momentum in correspondence of the excitation transducer and of the contact disc.](image-url)
A.1. Analytical model of the TRD

Referring to Figure A.1, the TRD can be divided into three sections of length $L_1$, $L_2$, $L_3$. Within each section, the behavior is described by the rotation angle $\theta_i(x_i, t)$ with $i = 1, 2, 3$. In each section, the general solution of Equation A.1 has the form:

$$\theta_i(x_i, t) = \hat{\theta}_i(x_i) e^{j\omega t} = (a_i \sin k_s x_i + b_i \cos k_s x_i) e^{j\omega t} \quad (A.5)$$

The six parameters $a_i$, $b_i$ for $i = 1, 2, 3$ must be solved by imposing the boundary conditions for each section, such as the continuity of the displacement field and the equilibrium equations (total of six equations). As an example, the equilibrium equation in correspondence with the electromagnetic transducer for excitation (see Figure A.2) is:

$$G^*_e J_p \theta_1, x(L_1) + J_m \ddot{\theta}_2(0) + d_s \dot{\theta}_2(0) = G^*_e J_p \theta_2, x(0) + M_e \quad (A.6)$$

The interaction with the viscoelastic medium on the contact disc is reported in Equation A.7 using the torsional impedance $Z_T$:

$$G^*_s J_p \theta_3, x(L_3) + J_d \ddot{\theta}_3(L_3) + Z_T \pi a^2 \dot{\theta}_3(L_3) = 0 \quad (A.7)$$

By writing all the six boundary conditions, the value of the coefficient $a_i$, $b_i$, and therefore of the rotations $\hat{\theta}_i$ can be found as a function of the input torque $\hat{M}_e$. The transfer function of the system can be defined as reported in Equation A.8:

$$\overline{TF}(\omega) = \frac{\theta_3(L_3, t)}{\hat{M}_e(t)} \quad (A.8)$$

The solution is not explicitly given here for space reason. A linear system of equations describes the behavior of the sensor, as in the matrix form of Equation A.9:

$$\mathbf{H}(\omega) \cdot \mathbf{a} = \mathbf{F}_e \quad (A.9)$$

where $\mathbf{a}$ is a vector that contains the coefficients $a_i$, $b_i$, $\mathbf{F}_e$ is a vector that contains the generalized external forces (in this case, the excitation torque $M_e$), and $\mathbf{H}(\omega)$ is a $6 \times 6$ matrix containing the information on the boundary conditions, and depends on the angular frequency $\omega$. For the TRD sensor, these matrix are:

$$\mathbf{a} = [a_1, b_1, a_2, b_2, a_3, b_3]^T$$

$$\mathbf{F}_e = [0, 0, M_e, 0, 0, 0]^T$$
A. Modeling the mechanical behavior of the sensors

\[ \mathbf{H}(\omega) = \begin{bmatrix}
0 & 1 & 0 & 0 & 0 & 0 \\
\sin(k_s L_1) & \cos(k_s L_1) & 0 & -1 & 0 & 0 \\
C_s \cos(k_s L_1) & -C_s \sin(k_s L_1) & -C_s & (j \omega d_s - J_m \omega^2) & 0 & 0 \\
0 & 0 & \sin(k_s L_2) & \cos(k_s L_2) & 0 & -1 \\
0 & 0 & C_s \cos(k_s L_2) & -C_s \sin(k_s L_2) & -C_s & -\omega^2 J_m \\
0 & 0 & 0 & 0 & A_{65} & A_{66}
\end{bmatrix} \]

where the following coefficients have been defined:

\[ C_s = G_s J_p k_s \]
\[ A_{65} = C_s \cos(k_s L_3) - \omega^2 J_{disc} \sin(k_s L_3) + j \omega Z_T \pi a^2 \sin(k_s L_3) \]
\[ A_{66} = -C_s \sin(k_s L_3) - \omega^2 J_{disc} \cos(k_s L_3) + j \omega Z_T \pi a^2 \cos(k_s L_3) \]

In Figure A.3, the transfer function of the system vibrating in air \((Z_T = 0)\) is reported for the range 1-12 kHz, showing the amplitude peaks and phase shifts of \(\phi = \pi\) corresponding to each torsional resonance frequency of the sensor. With the analytical model of the resonator described above, the experimental values of \(\Delta f_{res}\) and \(df\) obtained in a test with a viscoelastic medium can be converted into the torsional mechanical impedance \(Z_T\). To a specified value of \(Z_T\) corresponds a specific transfer function \(\hat{TF}(f, Z_T)\), from which the values of \(\Delta f_{res}\) and \(df\) can be evaluated for each resonance frequency. Figure A.4 shows the transfer function of the resonator for a typical input value of \(Z_T\).

**Figure A.3:** Transfer function of the TRD in the frequency range 1-12 kHz obtained with the analytical model. Logarithmic scale chosen for the amplitudes to emphasize the resonance peaks. Transfer function normalized to the static value \(TF_0\).

**Figure A.4:** Transfer functions of the vibrating system round the first resonance. Comparison between calibration in air and a measurement run with \(|Z_T| = 1.74 \cdot 10^{-2}\) and \(\phi = -0.104\). Expected values of \(\Delta f_{res} = 0.029\) Hz and \(df = 0.571\) Hz. These value correspond to \(G_1 = 17\) kPa and \(G_2 = 11.2\) kPa.
Eigenfrequencies and mode shapes

By setting the input torque $M_e = 0$, the system of six homogenous equations described in Equation A.9 admits an infinite number of solutions, each of those characterized by the eigenfrequency $\Omega^{(n)}$ that solve the eigenvalue problem. The associated coefficients $a_i^{(n)}$, $b_i^{(n)}$ can be used to determine the mode shapes of the sensor by the $n$-th eigenfrequency (in Figure 2.8 the first five mode shapes have been reported).

Intrinsic Damping

The intrinsic damping of a resonator is made by different components [44, 98]:

(i) material damping, previously treated introducing $G_s^*$,

(ii) coupling between the electrical circuit and the mechanical oscillator, since the moving magnets will produce currents in the surrounding conductive materials extracting energy from the resonator,

(iii) energy lost at the fixation, if the clamp is not properly designed,

(iv) energy lost in glued, screwed and brazed connections,

(v) acoustic radiation, if any moving parts have a component of the velocity normal to the surface.

Except (i), already modeled with the introduction of $G_s^*$, the other damping sources have been modeled introducing the lumped element with constant $d$. This constant must be determined with experiments at the different resonance frequencies of the resonator to tune the model to the real behavior.

Decoupling mass

To reduce the mechanical coupling between the torsional resonator and the surrounding world, a spring-mass system may be used, as sketched in Figure A.5. The decoupling mass consists of a mass element whose inertia is an order of magnitude higher than the resonating sensor. Between the decoupling mass and the fixation to the ground, a rubber styrofoam ring of torsional stiffness $c_{dec}$ is inserted.

The addition of a decoupling mass to the resonator has the effect to "force a node" in the system. The displacement of the root of the resonator, in correspondence with the decoupling mass, has a value that approaches zero. The torque $M_{fix}$ between the decoupling mass and the surrounding world, as depicted in Figure A.5, will also be reduced. Applying the angular momentum equation to the decoupling mass, it follows that:

$$-J_{dec} \omega^2 \theta_{dec} = -c_{dec} \cdot \theta_{dec} + M_s$$

(A.10)
A. Modeling the mechanical behavior of the sensors

\[ \theta_{\text{dec}} = \frac{M_s}{c_{\text{dec}} - J_{\text{dec}} \omega^2} \]  
(A.11)

\[ M_{\text{fix}} = c_{\text{dec}} \cdot \theta_{\text{dec}} = \frac{M_s}{1 - \frac{J_{\text{dec}} \omega^2}{c_{\text{dec}}}} \]  
(A.12)

\[ M_{\text{fix}} \ll M_s \text{ for } \omega^2 \gg \frac{c_{\text{dec}}}{J_{\text{dec}}} \]  
(A.13)

The torque applied to the surrounding world \( M_{\text{fix}} \) is strongly reduced with respect to the original torque \( M_s \) due to the vibration of the sensor, if the condition stated in Equation A.13 is verified. From the reduction of the torque \( M_{\text{fix}} \) benefits the quality factor of the device, since the intrinsic damping at the fixation is strongly reduced.

In the present arrangement, \( J_{\text{dec}} = 4.9 \cdot 10^{-4} \text{ kg} \cdot \text{m}^2 \), \( c_{\text{dec}} \approx 500 \text{ N} \cdot \text{m} \). For the first resonance frequency, \( \omega = 8.17 \cdot 10^3 \), and resulting in \( M_{\text{fix}} \approx 0.015M_s \).

![Scheme of the decoupling mass and rubber ring to reduce the mechanical coupling with the surrounding world.](image)

**Figure A.5:** Scheme of the decoupling mass and rubber ring to reduce the mechanical coupling with the surrounding world.
A.2 Analytical Model of the HFTR

The HFTR can be modeled analytically following the same approach outlined for the TRD. Figure A.6 shows the mechanical scheme of the device. The device is divided into two continuous domains (external tube case and internal rod), and studied with $\theta_1$ and $\theta_2$. Two polar inertia moments, $J_{p,ext}$ for the tube case and $J_{p,int}$ for the internal rod, respectively, must be considered. $G_s^*$ and $\rho_s$ identify the material parameters of the sensor, assumed to be the same for both external case and internal rod, characterized by the wave number $k_s$ previously defined in Equation A.2. The contact disc $J_d$, the magnet used for excitation $J_m$ and the decoupling mass $J_{dec}$ are modeled as discrete elements with concentrated inertias. The contact with the viscoelastic medium, characterized by the torsional impedance $Z_T$, takes place in correspondence to the contact disc, while the excitation torque $M_e$ is applied in correspondence to the magnet. The HFTR can be described by a system of linear equations (see Equation A.9) that results from the boundary conditions, where:

$$a = [a_1, b_1, a_2, b_2]^T$$

$$F_e = [0, 0, 0, M_e]^T$$

$$H(\omega) = \begin{bmatrix} C_{s,ext} \sin(k_s L_1) & J_{dec} \omega^2 & 0 & 0 \\ C_{s,ext} \cos(k_s L_1) & \cos(k_s L_1) & 0 & 0 \\ 0 & -C_{s,ext} \sin(k_s L_1) & -C_{s,int} & (-J_d \omega^2 + j\omega Z_T \pi a^2) \\ 0 & 0 & A_{43} & A_{44} \end{bmatrix}$$
A. Modeling the mechanical behavior of the sensors

where the following coefficients have been defined:

\[ C_{s,\text{ext}} = G_s^* J_{p,\text{ext}} k_s \]
\[ C_{s,\text{int}} = G_s^* J_{p,\text{int}} k_s \]
\[ A_{43} = C_{s,\text{int}} \cos(k_s L_2) - J_m^2 \omega^2 \sin(k_s L_2) \]
\[ A_{44} = -C_{s,\text{int}} \sin(k_s L_2) - J_m^2 \omega^2 \cos(k_s L_2) \]

The geometric and material parameters for the HFTR were reported in Figure 2.9.
Comparison with quasi-static tests

The results obtained with the dynamic torsion test for small deformations at high frequency have been compared in this thesis with those obtained from quasi-static experiments, capable of characterizing the materials at large deformations and at low frequencies. In Chapter 4, combined tests with the TRD and with the Aspiration Experiment of Nava and Mazza [61] on bovine liver tissues have been presented. In Chapter 5, the results obtained on EAPs with the dynamic torsion test at different levels of pre-strain have been compared to standard uniaxial tension tests.

The material samples considered were tested with quasi-static methods by applying large deformations, up to 30% nominal strain for the bovine liver tissue and up to 500% for the EAP. Their behavior was modeled using the theory of hyperelasticity, assuming quasi-linear viscoelasticity to reproduce the relaxation phenomenon.

In this appendix, a brief overview of the hyperelastic material models used to describe the behavior of soft biological tissues and EAP is given, reporting the material parameters extracted from the quasi-static experiments. The procedure used to compare these results with those obtained from dynamic torsion tests is also reported.

B.1 Hyperelastic material modeling

In continuum mechanics large strain elastic (so called hyperelastic) materials are characterized through a strain energy potential $W$ that represents the strain energy of the material as a function of the deformation. The following strain energy functions have been considered in this thesis:

(i) the reduced polynomial form [96], used to model the behavior of soft bovine liver tissue with the Aspiration Experiment [61], with

$$ W = \sum_{n=1}^{N} C_n (I_1 - 3)^n + \frac{1}{D}(J - 1)^2 \quad \text{(B.1)} $$
B. Comparison with quasi-static tests

where $I_1$ is the first invariant of the left Cauchy-Green deformation tensor, $J$ is the total volume change and $C_{n0}$ are the $N$ parameters ($n = 1, N$) that have to fitted from experiments. $D \to 0$ indicates a behavior tending to incompressibility, that is the case for most soft biological tissues consisting mainly of water. Nava and Mazza [61] used this model with $N = 5$ (fifth order) to model the behavior of soft biological tissues.

(ii) the Yeoh form [95] was used in the characterization of EAPs in large deformations with standard uniaxial tests, with

$$W = C_{10}(I_1 - 3) + C_{20}(I_1 - 3)^2 + C_{30}(I_1 - 3)^3$$

(B.2)

that is indeed a reduced polynomial form of the third order, with the three material parameters $C_{10}$, $C_{20}$ and $C_{30}$ that are determined through experiments.

The first invariant $I_1$ of the left Cauchy-Green deformation tensor depends on the principal stretch ratios $\lambda_i$ ($i = 1, 2, 3$) as

$$I_1 = \lambda_1^2 + \lambda_2^2 + \lambda_3^2$$

(B.3)

The stretch ratios $\lambda_i$ are defined as the current length divided by the original length in the principal directions of the deformation.

For a totally incompressible material, the nominal or engineering stress $s_i$ (the current force per unit area of the undeformed configuration) is given by the derivative of the strain energy potential with respect to the stretch ratio $\lambda_i$:

$$s_i = \frac{\partial W}{\partial \lambda_i} - \frac{1}{\lambda_i^p}$$

(B.4)

where $p$ is the hydrostatic pressure that depends on the kinetic boundary conditions. In a similar way, the true stress $t_i$ or Cauchy stress (the force vector per unit area in the present configuration, i.e. considering the change in the cross section of the sample), can be obtained as follows:

$$t_i = \lambda_i \frac{\partial W}{\partial \lambda_i} - p$$

(B.5)

Modeling viscoelasticity

The time dependence of the mechanical response has always been modeled in this work with the assumption of quasi-linear viscoelasticity [22]. In this formulation, the stresses in the material, which themselves may result from a nonlinear stress-strain relation, are linearly superposed with respect to time. The coefficients $C_{n0}$ of the strain energy potential are therefore time dependent.

One way to model this dependence is with the Prony series [85]. The hyperelastic material parameters $C_{n0}^R$, which include also the relaxation phenomenon in the
material, are defined as follows:

\[
C_{n0}^R(t) = C_{n0}^0 \cdot f(t) = C_{n0}^0 \cdot \left[ 1 - \sum_{k=1}^{K} g_k \cdot \left( 1 - \exp \left( -\frac{t}{\tau_k} \right) \right) \right]
\]  

(B.6)

where \(C_{n0}^0\) describe the instantaneous elastic response and \(g_k\) and \(\tau_k\) characterize the relaxation behavior with a set of \(K\) exponential Prony series. The function \(f(t)\) characterizes the relaxation behavior with \(f(t = 0) = 1\). Another formulation is also common in the literature, using the long-term elastic response \(C_{n0}^\infty\), that leads to:

\[
C_{n0}^R(t) = C_{n0}^\infty \cdot g(t) = C_{n0}^\infty \cdot \frac{1 - \sum_{k=1}^{K} g_k \cdot \left( 1 - \exp \left( -\frac{t}{\tau_k} \right) \right)}{1 - \sum_{k=1}^{K} g_k}
\]  

(B.7)

**Equivalent uniaxial curve and linearization**

In case of uniaxial tensile test (loading in direction 1), the nominal stresses in direction 2 and 3 are zero. Simple relations between the nominal stress and the stretch ratios can be calculated and used to fit experimental data. The nominal stress for the reduced polynomial form can be evaluated as:

\[
s_1 = 2 \left( \lambda_1 - \frac{1}{\lambda_1^2} \right) \sum_{n=1}^{N} nC_{n0} \left( \lambda^2 + 2\lambda^{-1} - 3 \right)^{n-1}
\]  

(B.8)

For the special case of the Yeoh form \((N = 3)\), follows that:

\[
s_1 = 2 \left( \lambda_1 - \frac{1}{\lambda_1} \right) \cdot \left[ C_{10} + 2 \cdot C_{20} \cdot \left( \lambda_1^2 + \frac{2}{\lambda_1} - 3 \right) + 3 \cdot C_{30} \cdot \left( \lambda_1^2 + \frac{2}{\lambda_1} - 3 \right)^2 \right]
\]  

(B.9)

In the last expression, the relation

\[
\lambda_2 = \lambda_3 = \frac{1}{\sqrt[3]{\lambda_1}}
\]  

(B.10)

has been used, which corresponds to an isochoric deformation \((\lambda_1\lambda_2\lambda_3 = 1)\). Equation B.9 can be used to determine the hyperelastic material parameters \(C_{10}, C_{20},\) and \(C_{30}\) from uniaxial test data.

Following the same procedure, the Cauchy stress can also be evaluated from B.8:

\[
t_1 = \lambda s_1 = 2 \left( \lambda_1^2 - \frac{1}{\lambda_1} \right) \sum_{n=1}^{N} nC_{n0} \left( \lambda^2 + 2\lambda^{-1} - 3 \right)^{n-1}
\]  

(B.11)
B. Comparison with quasi-static tests

The derivative of $t_1$ with respect to the stretch evaluated for an infinitesimal stretch ($\lambda \to 1$) corresponds to the instantaneous Elastic modulus of the material $E_0$. It can be shown that, for the reduced polynomial form (and therefore also for the Yeoh model):

$$E_0 = \frac{\partial t_1}{\partial \lambda_1} \mid_{\lambda_1\to 1} = 6C_{10} \quad \text{(B.12)}$$

$$G_0 = 2C_{10} \quad \text{(B.13)}$$

The value of the instantaneous shear modulus $G_0$ was obtained assuming incompressibility.

Frequency domain viscoelasticity

In order to compare the results obtained from the hyperelastic models with quasi-linear viscoelasticity to those obtained with the dynamic torsion test, it is necessary to convert the results obtained in the time domain with the quasi-static tests to the frequency domain.

The shear modulus relaxation function $G(t)$ will have the typical formulation described by the Prony series, as:

$$G(t) = G_0 \left[ 1 - \sum_{k=1}^{N} g_k \cdot \left( 1 - \exp \left( -\frac{t}{\tau_k} \right) \right) \right] \quad \text{(B.14)}$$

where $G_0$ is the instantaneous value and the coefficients $g_k$ and $\tau_k$ define the Prony series used. The complex shear modulus $G^*$ can be evaluated with Equation B.15:

$$G^*(\omega) = G(0) + \int_{0}^{\infty} e^{-j\omega\tau} \dot{G}(\tau) d\tau \quad \text{(B.15)}$$

From this equation, the two components $G_1(\omega)$ and $G_2(\omega)$, storage and loss modulus respectively, can be evaluated as follows:

$$G_1(\omega) = G_0 \left( 1 - \sum_{k=1}^{N} g_k \right) + G_0 \sum_{k=1}^{N} \frac{g_k(\omega\tau_k)^2}{1 + (\omega\tau_k)^2} \quad \text{(B.16)}$$

$$G_2(\omega) = G_0 \sum_{k=1}^{N} \frac{g_k\omega\tau_k}{1 + (\omega\tau_k)^2} \quad \text{(B.17)}$$
B.2 Aspiration experiments on bovine liver tissue ex-vivo

The Aspiration Device shown in Figure 4.22 has been developed by Vuskovic [91] and recently improved for new experiments by Nava and Mazza [61]. The device has been designed for in-vivo characterization of soft biological tissues. It consists of a tube in which the internal pressure can be controlled according to a desired pressure law. The experiment is performed by (i) gently pushing the tube against the tissue to ensure a good initial contact, and (ii) creating a (time variable) vacuum inside the tube so that the tissue is sucked in through the aspiration hole (diameter of 10 mm). A video camera grabs images of the deformation profile of the aspirated tissue, and a pressure sensor measures the correspondent vacuum level. The finite element method is employed in to solve the inverse problem: the experiment is simulated by a FE model in which the (time dependent) aspiration pressure is imposed as kinetic boundary condition; the material constants are determined iteratively from the comparison of calculated and measured soft tissue deformation [60], as reported in Figure B.1.

The aspiration device was used by Alessandro Nava, Institute of Mechanical Systems, ETH Zurich, to determine the mechanical properties of bovine liver tissue ex-vivo. An intact bovine liver was obtained from the slaughterhouse immediately following animal euthanasia. The liver was transported on ice and kept moist wrapped in a physiological saline soaked surgical cloth at 4°C until the samples were prepared. The experiments were performed on the intact organ, without removing the superficial membrane (capsule).

Figure B.1: Displacement of the top of the tissue bubble measured in the aspiration test (dotted line) and simulated by FE (continuous line).

Figure B.2: Equivalent uniaxial cauchy stress curve for bovine liver tissue samples tested ex-vivo. Curves presented with three different value of vertical force $F_v$. 

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B. Comparison with quasi-static tests

The liver tissue was modeled as an homogeneous and isotropic continuum, using a fifth order reduced polynomial form (see Equation B.1) for the strain energy function with quasi-linear viscoelasticity assumption. The results of these experiments are reported in Table B.1 for three different values of the contact force, which slightly influence the results [60]. In Figure B.2, the equivalent uniaxial curves evaluated using the fifth order reduced polynomial form are reported (instantaneous values). By taking the derivative of these curves in correspondence of $\lambda=1$, the instantaneous elastic modulus $E_0$ can be evaluated.

Table B.1: Material constants for the fifth order reduced polynomial form model extracted from the Aspiration Experiment [61] on bovine liver tissue ex-vivo. The viscoelastic behavior is modeled with Prony series. Results are given for three different values of the contact force $F_v$. Courtesy of Alessandro Nava [60].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>$F_v$=5N</th>
<th>$F_v$=3.5N</th>
<th>$F_v$=2.5N</th>
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<tbody>
<tr>
<td>$C_{10}^\infty$ [kPa]</td>
<td>8.21</td>
<td>7.33</td>
<td>5.22</td>
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<tr>
<td>$C_{20}^\infty$ [kPa]</td>
<td>8.04</td>
<td>9.03</td>
<td>9.84</td>
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<tr>
<td>$C_{30}^\infty$ [kPa]</td>
<td>8.16</td>
<td>7.42</td>
<td>7.84</td>
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<td>$C_{40}^\infty$ [kPa]</td>
<td>26.83</td>
<td>29.10</td>
<td>29.41</td>
</tr>
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<td>$C_{50}^\infty$ [kPa]</td>
<td>44.77</td>
<td>46.71</td>
<td>49.23</td>
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<tr>
<td>$g_1$ [-]</td>
<td>0.29</td>
<td>0.26</td>
<td>0.27</td>
</tr>
<tr>
<td>$\tau_1$ [s]</td>
<td>2.73</td>
<td>3.75</td>
<td>3.80</td>
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<tr>
<td>$g_2$ [-]</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
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<tr>
<td>$\tau_2$ [s]</td>
<td>20.06</td>
<td>20.48</td>
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<tr>
<td>$g_3$ [-]</td>
<td>0.09</td>
<td>0.09</td>
<td>0.09</td>
</tr>
<tr>
<td>$\tau_3$ [s]</td>
<td>27.51</td>
<td>29.25</td>
<td>30.10</td>
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<tr>
<td>$g_4$ [-]</td>
<td>0.10</td>
<td>0.10</td>
<td>0.11</td>
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<tr>
<td>$\tau_4$ [s]</td>
<td>154.0</td>
<td>147.5</td>
<td>154.5</td>
</tr>
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</table>

Assuming incompressibility, $G_0 = E_0/3$. From the shear relaxation modulus $G(t)$, obtained as in Equation B.14 with Prony series, the complex shear modulus $G^*$ can be evaluated using Equation B.15. Figure B.3 shows the linearized behavior evaluated from the aspiration tests for the three different values of the vertical force $F_v$, using the parameters reported in Table B.1. The results obtained with the Aspiration Device can be considered reliable up to a frequency of 0.1 Hz due to the characteristics of the experiment and to the type of pressure law applied.
B.3 Uniaxial tests for the dielectric polymer VHB 4910

The dielectric polymer VHB 4910 was characterized at the EMPA, by Michael Wissler [93]. Uniaxial tensile tests have been carried out with a Zwick Z010 machine (Zwick Roell, Ulm, Germany) on specimens with undeformed dimensions of 150mm×10mm×1mm (length×width×thickness).

Uniaxial relaxation experiments were performed at room temperature by stretching the samples to a predefined elongation and measuring the force decrease over a time period of approximately 0.5 h. Fourteen experiments have been performed, the nominal strain being between 20% and 500%. Examples of the relaxation curves obtained from these experiments are reported in Figure B.4 for three values of the pre-stretch. Uniaxial tensile tests have been performed for two samples with a Zwick (Z010) machine at room temperature: the specimens were loaded up to 300% nominal strain linearly over a time period of 900 s, the strain was hold fix for 900 s, and the sample was than unloaded with a negative strain rate of -0.33% s$^{-1}$. Figure B.5 shows the results of this experiment.

The hyperelastic type of response of the material was modeled using the strain energy potential of Yeoh [95], see Equation B.2, using Prony series to describe the time dependence of the mechanical response. The results of the curve fit is reported in both Figures B.4 and B.5. Table B.2 reports the value of the coefficients used in the hyperelastic model.
B. Comparison with quasi-static tests

![Figure B.4](image1.png)\[Image: Relaxation curves for the VHB 4910 at different levels of the pre-stretch.\]

![Figure B.5](image2.png)\[Image: Tensile tests on two specimens of VHB 4910. A trapezoidal time history for the strain were applied to the specimens.\]

Table B.2: Material constants for the Yeoh model obtained for the VHB 4910. Courtesy of Michael Wissler [93].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tr>
<td>$C_{10}^0$ [MPa]</td>
<td>0.0693</td>
</tr>
<tr>
<td>$C_{20}^0$ [MPa]</td>
<td>$-8.88 \times 10^{-4}$</td>
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<td>$C_{30}^0$ [MPa]</td>
<td>$16.7 \times 10^{-6}$</td>
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<td>$g_1$ [-]</td>
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<tr>
<td>$\tau_1$ [s]</td>
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<td>$g_2$ [-]</td>
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<tr>
<td>$\tau_2$ [s]</td>
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<tr>
<td>$g_3$ [-]</td>
<td>0.110</td>
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<td>$\tau_3$ [s]</td>
<td>37.1</td>
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<tr>
<td>$g_4$ [-]</td>
<td>0.0384</td>
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<td>$\tau_4$ [s]</td>
<td>217</td>
</tr>
</tbody>
</table>

In Figure B.6, the uniaxial instantaneous curve for the nominal stresses and for the true (Cauchy) stress is reported. From the true stress curve $t_1(\lambda_1)$, the tangent instantaneous elastic modulus $E_0(\lambda)$ for a given stretch $\lambda$ can be extracted as:

$$E_0(\lambda) = \frac{\partial t_1}{\partial \lambda_1}\bigg|_{\lambda_1=\lambda} \quad \text{(B.18)}$$

Figure B.7 shows the behavior of $E_0(\lambda)$ evaluated from the Yeoh model. As evident from Figure B.7, the tangent modulus increases with increasing stretch.
B.3. Uniaxial tests for the dielectric polymer VHB 4910

Figure B.6: Uniaxial instantaneous hyperelastic response: Yeoh model obtained from uniaxial experiments. Nominal and Cauchy stresses as a function of the uniaxial stretch λ.

Figure B.7: Instantaneous tangent elastic modulus $E_0(\lambda)$ obtained from the Yeoh model of the VHB 4910.

Assuming incompressibility, the tangent elastic modulus can be linked to the tangent shear modulus $G_{0t}$ simply by dividing by a factor of 3:

$$G_0(\lambda) = \frac{E_0(\lambda)}{3}$$  \hspace{1cm} (B.19)

The viscoelastic behavior is introduced with Prony series,

$$G(t) = G_0(\lambda) \left[ 1 - \sum_{k=1}^{N} g_k \cdot \left( 1 - \exp \left( -\frac{t}{\tau_k} \right) \right) \right]$$  \hspace{1cm} (B.20)

where $G_0(\lambda)$ is the instantaneous value (dependent on the pre-stretch $\lambda$). Considering the case of $\lambda = 1$, the storage and loss module, $G_1(\lambda = 1)$ and $G_2(\lambda = 1)$ can be evaluated in the frequency domain using Equations B.16 and B.17. Figure B.8 shows the corresponding curves in the low frequency domain. Similar curves can be obtained for different values of the pre-stretch $\lambda$.

The results can be considered reliable up to a frequency of 1 Hz due to the type of experiments used to obtain the material constant. DMA tests at higher frequencies [81] must be carried out to to extend the material characterization above 1 Hz.
B. Comparison with quasi-static tests

Figure B.8: Linearized viscoelastic behavior of the VHB 4910 in the frequency domain: $|G^*|$ and $\delta$ for $\lambda = 1$. 
Bibliography


Curriculum Vitae

Davide Valtorta
Born on January 11, 1976 in Cantù, Italy
Citizen of Italy

Education

<table>
<thead>
<tr>
<th>Dates</th>
<th>Institution and Program</th>
</tr>
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<tbody>
<tr>
<td>Sep. 1982 - Jun. 1990</td>
<td>Primary and Secondary school, Cucciago, Italy</td>
</tr>
<tr>
<td>Sep. 1990 - Jul. 1995</td>
<td>High School, Liceo Scientifico Enrico Fermi, Cantù, Italy</td>
</tr>
<tr>
<td>May 2002 - Sep. 2006</td>
<td>Doctoral student at the Institute of Mechanical Systems, ETH Zurich, Zurich, Switzerland</td>
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Professional Experience

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<th>Dates</th>
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<tbody>
<tr>
<td>Sep. 2000 - Jun. 2001</td>
<td>Research Assistant in railway vehicle dynamics, University of Illinois, Chicago, USA</td>
</tr>
<tr>
<td>Jan. 2002 - May 2002</td>
<td>Project Engineer, Electrolux Home Products, Solaro, Italy</td>
</tr>
<tr>
<td>May 2002 - Sep. 2006</td>
<td>Teaching Assistant at the Institute of Mechanical Systems, ETH Zurich, Zurich, Switzerland</td>
</tr>
<tr>
<td>since Nov. 2006</td>
<td>Consultant in design process innovation, CADFEM AG, Aadorf, Switzerland</td>
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Contributions to conferences


Peer-reviewed journal papers


