PART I

Mechanical and Electronic Instrumentation

Chapter 1

Guidelines for Choosing the Experimental Set-up

From an experimental point of view, the elastic and/or viscoelastic characterization of materials is not necessarily achieved simply by using an existing piece of industrial apparatus.

To begin with, the researcher has to choose the experimental set-up, taking the following items into account:

– the type of wave, whether progressive or stationary;

– the measurement technique;

– the numerical method to calculate the elastic (or viscoelastic) modulus or stiffness coefficient.

In this chapter, choice criteria as well as selection guidelines are presented. The following topics will be discussed in turn:

– choice of matrix coefficient(s) (stiffness or compliance matrix) to be evaluated;

– frequency range in which tests are to be conducted;

– shape and dimensions of the sample;

– temperature range to be adopted;

– viscoelastic properties of the material frequency dependence, damping capacity, etc.

Chapter written by Jean Tuong VINH

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– available previsional calculations (for composite materials) which enable the order, or the range, of elastic constants to be obtained.

1.1. Choice of matrix coefficient to be evaluated and type of wave to be adopted

1.1.1. *For isotropic materials*

The number of elastic constants is reduced to two, chosen from five available elastic constants: Young's modulus, E; shear modulus, G; Poisson's number, ν; volumic dilatation, K; and the stiffness coefficient C_{iiii} related to an extensional wave. For mechanical applications at low and medium frequency range ($f \leq 10{,}000$ Hz), a compliance matrix [S] is preferred.

Isotropic material tests			
Stationary waves in rods		Progressive waves in thick plates	
Young's modulus E or E_{ii}	Shear modulus G or G_{ij} ,($i \neq j$)	Ultrasonic waves	
Bending wave or extensional wave	Torsional wave	Extensional waves C_{iiii}	Shear waves C_{ijij} , $i \neq j$

Table 1.1. *The two classes of tests to be selected when the text material is isotropic*

In Table 1.1 the two classes of tests[1] permitting the evaluation of a compliance matrix [S] and a stiffness matrix [C] are presented. A bending wave enables the Young's modulus to be obtained, and a torsional wave, the shear modulus. A bending wave is preferred to an extensional wave for many practical reasons[2]:

– the ease with which measurements are effected;

– a bending wave dispersion is completely portrayed by the fourth order equation of motion (Mindlin–Timoshenko's equation or, with restriction at lower a frequency range, Bernoulli–Euler's equation);

– an extensional wave is the other possibility. However, at medium and higher frequency ranges, a sixth order equation of motion is referred to and consequently it is more difficult to handle the characteristic functions.

The ultrasonic method is easy to carry out. A thick plate sample must be chosen so as to produce, with some care, progressive waves (extensional or shear) in the samples. The wavelength Λ through thickness h satisfies the following inequality:

1 Indexes are used for anisotropic composite materials and not for isotropic materials.
2 The wave dispersion of an extensional wave requires a sixth order equation of motion to cover the whole frequency range.

$$\Lambda / h \leq 4 \text{ to } 5 \qquad [1.1]$$

1.1.2. *For anisotropic materials*

The number of elastic constants depends on the degree of symmetry of the material. Remember that the number of different constants required for various materials is as follows:

– orthotropic material (wood): 9 constants;

– quasi-transverse (tetragonal) material: 6 constants;

– transverse isotropic material (long fibers regularly distributed in resin matrix): 5 constants;

– quasi-isotropic (cubic) material: 3 constants.

If preliminary information about the material is known (for example the degree of material symmetry [CHE 10]), the samples (number and shape) can be tailored with respect to the symmetry axis of the material. For a rod sample, its axis can be chosen to be coincident or different from the axis of symmetry of the material. For plates in ultrasonic tests, the material axis of symmetry may be collinear (or not) with the plate axis along the thickness, and the propagation direction of waves in any direction is obtained by transducer orientations.

1.1.2.1. *Orthotropic material*

Figure 1.1 shows three rods (of rectangular or square section) which are fabricated with a rod axis collinear with one material axis. From these three samples, six compliance matrix coefficients can be obtained:

$$S_{1111} = S_{11} = 1/E_1, \quad S_{2222} = S_{22} = 1/E_2, S_{3333} = S_{33} = 1/E_3 \qquad [1.2]$$

$$4S_{2323} = S_{44} = 1/G_{23}, 4S_{3131} = S_{55} = 1/G_{31}, 4S_{1212} = S_{66} = 1/G_{12}\text{[3]} \qquad [1.3]$$

Three remaining non-diagonal coefficients are to be evaluated. For this purpose, three other rod samples are fabricated. These samples are off-axis rods. The angles formed by the rod axis and the reference axis related to the material must be optimized, as in Figure 1.1(b).

The three off-axis samples permit the three non-diagonal compliance coefficients to be evaluated.

3 Figures in subscript are different in tensorial and matrix notations. For shear moduli G_{ij}, subscripts i and j indicate the plane in which shear stress and strain occur.

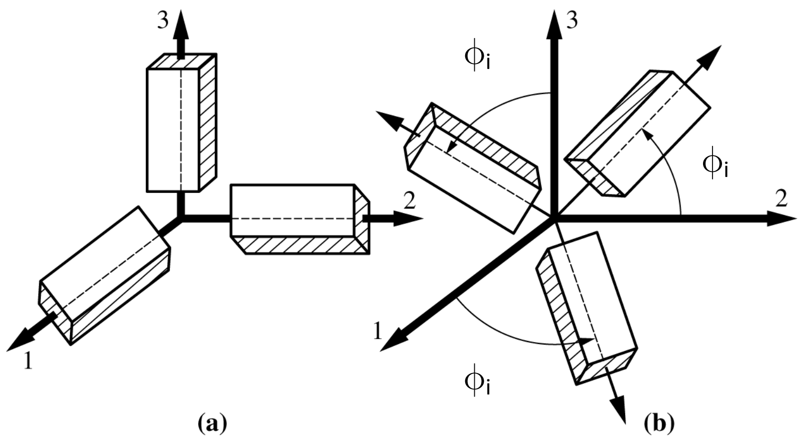

Figure 1.1. *Orthotropic material samples to be fabricated: a) three rods whose axes are respectively collinear with one of the symmetry axis of the orthotropic material. b) Off-axis rods whose axes are in the planes (1, 2), (2, 3), (3, 1) delimited by the symmetry axes of the material. Rod axes make angles $\phi_i \neq 0$, i= (1, 2, 3)*

Attention should be focused on the accuracy of the angles ϕ_i with which the rod samples in Figure 1.1(b) must be made. As the rod axes do not coincide with the symmetry axes of the material, we have to deal with the change of reference axes for tensor components of the fourth order in formulae giving non-diagonal compliance coefficients, since a weak variation of angle ϕ_I gives rise to an important variation of power four (see [CHE 10] Chapter 1, pp. 29-31) of the trigonometric functions. For plates tailored for ultrasonic measurement, we have to deal with a Christoffel's tensor of power 2: the accuracy of the angle ϕ formed between the normal coordinate system tied up to the plate sample and one of the symmetry axes of the material intervenes in a Christoffel's tensor of power 2, (see [CHE 10] Chapter 10, pp. 517-523) consequently this influence is less "critical" than the aforementioned compliance matrix coefficients.

1.1.2.2. *Transverse isotropic material*

For artificial composite materials, a transverse isotropic symmetry is usually adopted [CHE 10]. This involves uniaxial long fibers periodically distributed in a resin matrix. Figure 1.2 presents two rod samples whose z axes are, respectively, collinear with symmetry axis 3 and an off-axis rod, whose z axis makes an angle θ with the material plane (1, 2). The third sample is an off-axis whose z axis makes an angle $\theta \neq 0$ with material axis 3. The two first samples, 1 and 2, enable calculations of the following compliance coefficients:

$$S_{1111} = S_{11} = 1/E_1 = 1/E_2, \quad S_{3333} = S_{33} = 1/E_3 \qquad [1.4a]$$

$$4S_{1212} = S_{66} = 1/G_{12}$$

$$4S_{2323} = 4S_{1313} = S_{44} = S_{55} = 1/G_{13} = 1/G_{23} \qquad [1.4b]$$

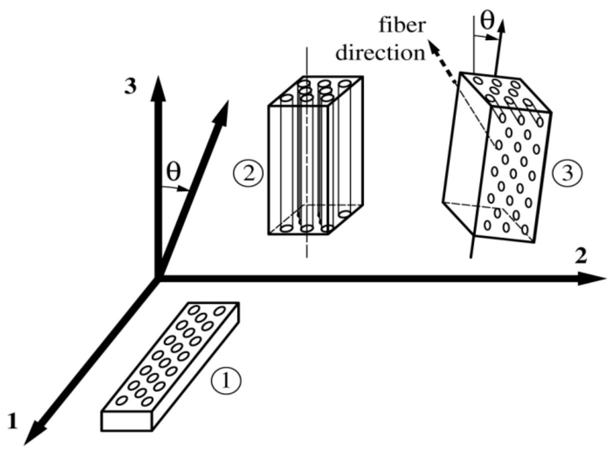

Figure 1.2. *Transverse isotropic material with. the material isotropic plane (1, 2) and material symmetry axis 3. Samples 1 and 2 allow four independent compliance coefficients to be obtained. Sample 3 enables calculation of* S_{23}

The third sample allows a material non-diagonal coefficient to be obtained and consequently Poisson's numbers ν_{32} and ν_{23}

$$S_{1133}=S_{2233}=S_{13}=S_{23}= -\nu_{32}/S_{22}= -\nu_{32}/S_{33} \qquad [1.5]$$

The samples used in Figure 1.2 are fragile during fabrication, as well as during measurement manipulation:

– for sample 3, the accuracy of the non-diagonal compliance coefficient strongly depends on the accuracy of the angle θ;

– if the material is strongly viscoelastic (i.e. if the damping capacity is high (tanδ)), the experimenter must be careful when using both the vibration and ultrasonic progressive wave techniques. Results obtained from the two techniques cannot be used concurrently to evaluate the remaining matrix coefficients. Since the working frequencies of waves are not in the same frequency range, the calculations might give rise to large errors.

The second reason to avoid this "mixing" method is that stationary waves require the use of a compliance matrix even though ultrasonic progressive waves concern a stiffness matrix. Matrix inversion is possible if a complete set of experimental stiffness coefficients has already been obtained.

Table 1.2 shows the two classes of testing methods. Details concerning dimensions of samples will be discussed later.

Anisotropic material (Degree of material symmetry Number of coefficients to be determined)					
Stationary waves in rods			Progressive waves in plates		
Bending vibration	Torsional vibration	Coupled vibrations	Extensional wave	Shear wave	Coupled waves
S_{11} ,S_{22},S_{33}	S_{44}, S_{55},S_{66}	S_{31}, or n_{31}	$C_{1111}= C_{11}$ $C_{3333}= C_{33}$	$C_{2323} = C_{44}$ $C_{1212} = C_{66}$	$C_{1133} = C_{13}$

Table 1.2. *For transverse isotropic material, five stiffness (or compliance) coefficients should be determined. The two classes of testing methods are used concurrently*

1.1.2.3. *Orthotropic materials*

For ultrasonic testing of massive materials, such as wood[4] or artificial three-dimensional composites, three thick plates can be cut (one with an axis collinear with the material's symmetry axis (or trunk axis): a radial plate; one plate at the peripheral; and a tangential plate, perpendicular to material symmetry axis) although difficulty exists in fabricating off-axis plates. For vibration tests on rods, readers should consult Figure 1.1.

1.2. Influence of frequency range

This question is related to the viscoelastic behavior of material. The following remarks might be helpful for experimenters.

4 The elastic properties of wood from the center of a trunk to the bark might present a gradient in mechanical properties which constitutes a particular problem to be solved.

1.2.1. *The Williams-Landel-Ferry method*

It is useful to use the William-Landel-Ferry method to obtain artificial enlargement of the frequency range by using temperature as a variable parameter. The applicability of this method (presented in detail elsewhere: see [CHE 10], Chapter 10) must be valid. It concerns the correspondence (temperature-frequency) principle.

1.2.1.1. *Adjustable temperature*

The use of a climatic chamber with positive and negative temperature adjustments is appropriate. A gradient of temperature on the rod is, however, to be avoided if time delay is not respected for temperature stabilization during heating or cooling operation.

1.2.1.2. *Choice of frequency range*

If a narrow frequency range is adopted, the dimensions of the samples should be chosen so as to obtain measurable amplitude of vibration on the sample. Attention should be focused on the first resonance frequencies.

The choice of frequency range is closely related to wave dispersion. Precaution should be taken to evaluate the wave dispersion correctly before evaluating the viscoelastic dispersion.

1.2.1.3. *Ultrasonic tests*

The working frequency is that of the transducer itself; it should correspond to the frequency (resonance frequency) of the transducer. The temperature is generally the ambient temperature, except in the case where the transducer coupling medium between the transducer and the sample is not a liquid coupling but a special long rod, at the end of which the sample is glued. The sample can be in a special chamber at high temperature, T > 100°C.

1.3. Dimensions and shape of the samples

If a large volume of material is at the experimenter’s disposal, plates and rods can be easily fabricated. However, some rod shapes are better suited to testing and calculations. The following practical considerations are useful.

1.3.1. *Square section rod for longitudinal wave*

If an extensional wave is adopted, a square section is to be preferred to a rectangular section. The reason for this is that the dispersion curve (velocity versus wave number or frequency) is less pronounced for a square section than in the case of a rectangular section, with flatness coefficient b (width)/h (thickness) < 1.

1.3.2. *Rod slenderness*

Rod slenderness is defined as the ratio h (thickness)/L (length). If the smallest possible slenderness is chosen, a large number of resonance frequencies is obtained. Higher frequencies are thus more easily obtained.

1.3.3. *Imposed shape and size*

In many cases it is difficult to obtain the shape and size wished for. In tests on bone, for example, the sample may be small in size, with a curved section. There is no possibility of cutting from a larger sample and one cannot manufacture a flat rod sample. One knows that there is a gradient in the elastic properties from the bone center to the free surface. In this case, the ultrasonic technique with special transducers would be the best method to adopt. Curved samples are often imposed on the experimenter (see [CHE 10], Chapter 10).

1.4. Tests at high and low temperature

Elastic and/or viscoelastic properties of materials change with temperature. A temperature controlled room with adjustable temperature between about -70°C to 250°C would be useful. No temperature gradient on the sample should be accepted. For negative low temperatures, a forced preliminary heating ventilation would be useful to avoid ice condensation on the sample.

Special transducers with special connecting cables are necessary for high temperatures.

1.5. Sample holder at high temperature

The sample holder plays an important role. Caution must be taken in bolt and screw systems to maintain the sample firmly without deforming the sample at the contact zones between sample and holder. Clamping systems require special precautions so as to avoid material creep at high temperatures.

1.6. Visual observation inside the ambient room

A glass window is necessary to examine the sample during tests. The window must be able to withstand high temperature.

1.7. Complex moduli of viscoelastic materials and damping capacity measurements

Measurement techniques deserve the special attention of the experimenter. Measurement techniques change drastically depending on the magnitude of the material damping capacity. With a very low damping coefficient of tan $\delta \approx 10^{-3}$ measurement at ambient atmosphere is subjected to large errors. The first factor to take into account is air damping around the sample, which is of this order of the material's damping tanδ in the interval (10^{-3}-5.10^{-3}). To avoid this disadvantage, a special set-up with a vacuum system is necessary. Damping of the sample holder is also to be taken into account. Precautions concerning measurement techniques will be examined in Chapter 8.

1.8. Previsional calculation of composite materials

Surprisingly, at first sight, this topic is presented as a useful companion to testing. It merits some explanation. In dynamic tests, the experimenter is often confronted with a problem of the magnitude of the first eigenfrequency, the dimensions and size of the sample being known before a test. If the order of elastic moduli is known in advance it will be a great help for the experimenter to choose the frequency range and to possibly discard resonance frequencies due to parasitic oscillations of the sample holder system and the exciter (see [CHE 10], Chapter 1).

The topics presented above constitute only preliminaries which will be expanded in the following chapters.

1.9. Bibliography

[CHE 10] CHEVALIER, Y., and VINH, J.T. (ed.), *Mechanics of Viscoelastic Materials and Wave Dispersion*, ISTE Ltd, London and John Wiley & Sons, New York, 2010.