Chapter 2

Review of Industrial Analyzers for Material Characterization

The issue of instrumentation for material characterization is indeed a very wide subject, covering numerous different areas of concern, such as:

– the mechanical behavior of high polymers;

– the elaboration of artificial composite materials;

– metallic materials for special applications;

– the application of materials in special conditions of temperature and environment;

– the characterization of materials in a large range of temperature and/or frequencies, in view of special applications requiring quantitative information on the viscoelastic behavior of materials;

– correlation between mechanical viscoelastic properties of materials and molecular interpretation, as well as analysis of structure geometry.

There are many books in which the principles of measurement of each of these apparatus are presented in detail, including those by J.D. Ferry [FER 69] and L.E. Nielsen [NIE 74], and I.M. Ward [WAR 71], in a series of chapters, presented a comprehensive discussion of the last item in the list above.

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Mechanical Characterization of Materials and Wave Dispersion: Instrumentation and Experiment Interpretation
Edited by Yvon Chevalier and Jean Tuong Vinh

With this book being oriented towards mechanical applications, the mechanical point of view is consequently emphasized. The review of some available industrial analyzers enables the reader to appreciate the mechanical conception of each instrument and understand whether it is suitable for their needs. Often, the mechanical part of the instrument is presented in such a manner that it is hidden or drowned in a complex whole, where the electronic equipment and automatically programmed calculations by computers seem to be the most important part of the analyzer.

Often, the first-time user of this kind of instrument can feel that it is comfortable and easy to use, and almost forgets to ask the main question: is the instrument well adapted for my measurement objectives?

2.1. Rheovibron and its successive versions

2.1.1. *Testing of filamentous sample and short rods*

Rheovibrons such as DMA 100, 150, 400 (Dynamic Mechanical Analyzers) or VHF 104 allow the measurement of the complex Young's modulus of samples presented as filaments. Figure 2.1 presents the principles of the apparatus. The sample filament is placed in a climatic chamber whose temperature can be adjusted in the range -150°C < T < 300°C.

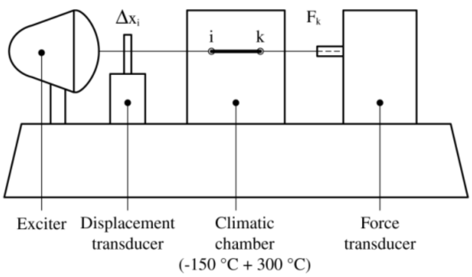

Figure 2.1. *Schematic diagram of a Rheovibron*

The forced vibration imposed on the sample is produced by an electromagnetic exciter (on the left of the diagram in Figure 2.1). At the entrance to the oven, a displacement strain gauge transducer measures Δx imposed to the point i of the filament. The tip k at the right of the sample is connected to a force strain gauge transducer which measures the force F_k. These two transducers give, through two Wheatstone bridges, two electrical tensions E_d and E_f such as:

$$e_d = (k_d E_d)\,\Delta x \qquad [2.1a]$$

$$e_F = (k_F E_F)\,F \qquad [2.1b]$$

$K_{d,}$ E_d, $K_{F,}$ E_F are proportional constants of the apparatus related to the measurement circuits. From [2.1a] and [2.1b] longitudinal strain and stress are evaluated:

$$\varepsilon^* = \Delta x / L = (e_d / k_d E_d L)$$

$$\sigma^{**} = F^*(\text{force})/S(\text{area of the filament cross-section}) \qquad [2.2]$$

Stars designate complex quantities.

Bringing [2.1] into [2.2], one obtains the complex Young's modulus E^*:

$$\sigma^* / \varepsilon^* = (e_F/e_d)\,(k_d/k_F)\,(E_d^* / E_F^*)\,(L/S) = E^* \qquad [2.3]$$

The first parenthesis corresponds to a deviation of an electronic instrument, the second and the third parentheses are apparatus constants, and the fourth parenthesis is related to the sample. As the measurement is effected under harmonic regime, there is a phase angle between the two vectors σ^* and ε^*.

An electric phasemeter permits the measurement of the phase angle between stress and strain. If the electric voltage proportional to σ^* is adjusted so that V_σ equals the tension proportional to ε^*:

$$|\,k_\sigma \sigma^*| = |\,k_\varepsilon \varepsilon^*| = V \qquad [2.4]$$

Figure 2.2 shows that the vector joining the two vectors is equal to 2V sin $(\delta_E/2)$.

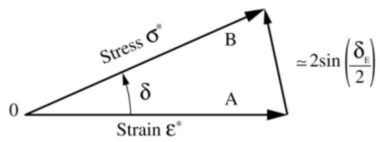

Figure 2.2. *Measurement of the phase angle δ. Vector B is electrically adjusted so that its absolute value equals the absolute value of vector A*

Conversion of sin (δ/2) into tan δ is possible:

$$\tan \delta = \tan(2 \text{ Arc} \sin(\delta/2) \quad [2.5]$$

The apparatus can accommodate a fiber of length $2 < L < 6$ mm, with a sample cross-section area $s < 0.2$ cm^2. Since in [2.5] the complex Young's modulus is measured in gain and phase, (E*, δ), the real and imaginary parts of the Young's modulus can be evaluated:

$$\text{Real } E^* = E' = |E^*| \cos \delta$$

$$\text{Imaginary } E^* = E'' = |E^*| \sin \delta \quad [2.6]$$

The direct reading of tan δ and the absolute value of E* on the one hand, and the wide temperature range on the other hand, are the main characteristics of this instrument. The measured loss coefficient tan δ is in the range:

$$10^{-3} < \tan \delta < 1.7 \quad [2.7]$$

2.1.2. *Improvement of the Rheovibron: the Rheovibron viscoanalyzer DDV II*

The initial conception of the mechanical part of the Rheovibron presents many weak points, detailed as follows:

– the elastic compliance of the mechanical system must be taken into account, including the exciter itself and the connection with the sample, as well as the connection between the sample and the stress gauge;

– at high temperature, samples tend to yield between grips. Correction is necessary;

– Massa [MAS 73] suggested mechanical improvements for the inertia of the mechanical system, and proposed correction factors for varying temperature and frequency as well as for the dimensions of the samples themselves, and even for the nature of the material.

This raises the problem of direct connection of the sample via sample holders and the mechanical environment, which is difficult to introduce into the equation given the complex Young's modulus, and complex additional dynamic systems (see Figure 2.3). These last set of mechanical components are made up of spring, dashpot (which are frequency dependent and temperature dependent) and inertia of exciter and transducer.

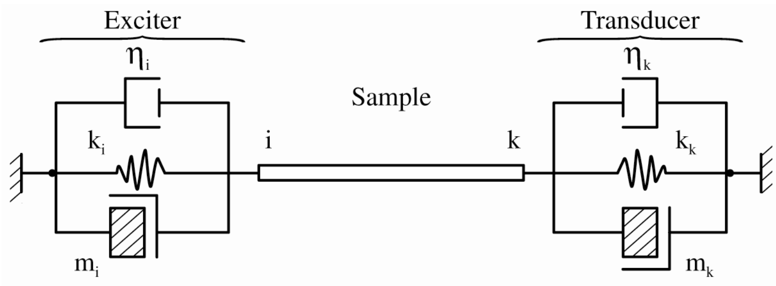

Figure 2.3. *Having an exciter and a mechanical sample holder introduces two sets of components on each side of the sample, constituted by spring k_i, dashpot η_i and mass m_i*

Modification of the sample holders has been proposed to adapt the instrument for bending tests. Erhard [ERH 70] suggested new holders for shearing tests. Compression tests and bending tests were proposed by Murayama [MUR 67] for measurements on anisotropic materials.

A servo-hydraulic actuator was introduced to increase the dynamic load capacity in magnitude as well as in a lower frequency range (f ≅ 0.1 to 5Hz), and a piezoelectric transducer was introduced for force measurements.

2.1.3. *Automated and improved version of Rheovibron by Princeton Applied Research Model 129 A*

This model, an automated Rheovibron by Princeton Applied Research, introduces two phase locks in the amplifier and data logging system.

2.2. Dynamic mechanical analyzer DMA 01dB – Metravib and VHF 104 Metravib analyzer

This French apparatus is initially adapted for short samples working in dynamic compression tests. Figure 2.4 shows a material presented as a short cylinder between two masses, m_1 and m_2. The mechanical holder and sample work as a two degrees of freedom system. The frequency response of the system presents a resonance and anti-resonance. The useful frequency part of the system is between these two extremes. The complex stiffness k^* of the sample, magnitude $|k^*(\omega)|$ and the phase (damping) angle δ_E are evaluated:

$$k^*(\omega) = \mathscr{R}e[\, k^*(\omega)] + j\mathfrak{I}m\, [k^*(\omega)]$$

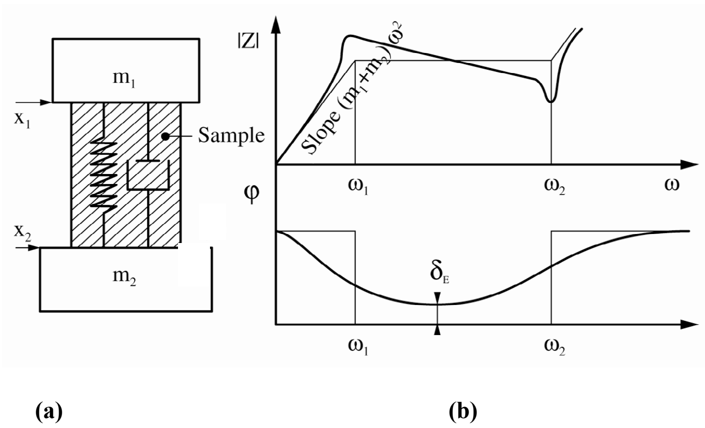

Figure 2.4. *A 01dB Metravib instrument: (a) schematic diagram of loading system and displacement measurements x_1 and x_2; (b) gain and phase response of the system, presenting resonance and anti-resonance. The working frequency interval is between the two zones. $|Z|$ is the transfer function x_2^*/x_1^* and φ is the phase angle of the whole system (the sample and additional masses)*

2.2.1. *Comments*

To avoid buckling, a sample working in compression must be short, which raises the question of three-dimensional stress states in the sample. The influence of shear stress as well as the influence of friction at both ends of the sample between the two masses m_1, m_2 and the sample must be taken into account.

Let us mention that the 01dB Metravib manufacturer proposes a large range of analyzers with a complete set of sample holders which allows compression, tension, bending tests as well as torsion and shearing tests with a variety of shapes and dimensions of samples. In the domain of mechanical analyzers with VHF apparatus, the frequency interval is extended from 100 Hz to 10 kHz. Depending on the adopted version, the ambient temperature of the sample can be chosen from between -50 °C and +110 °C.

2.3. Bruel and Kjaer complex modulus apparatus (Oberst Apparatus)

Bruel and Kjaer, a Danish company, specializes in electronic dynamic measurements (frequency spectrometers, frequency oscillators and level recorders). This company proposes a mechanical system in which the sample is presented as a vibrating reed working in forced flexural vibrations, also referred to as an “Oberst apparatus”.

The sample is clamped at one end (Figure 2.5). A force transducer also working as a force exciter is located at the lower end. The displacement transducer is a contactless one, located at a point between the clamping ends.

Measurements of the flexural Young's modulus $|E^*|$ and damping tan δ_E are effected at and around resonance frequencies of the sample:

$$E^* = \rho S \omega^2 L^4 / \beta^{4I} \quad [2.8]$$

where ρ is the density, S the area of the cross-section, ω the circular frequency, L the sample length, I inertia of the section in bending ($I = bh^3/ 12$), b width, h thickness and β^* the root of the eigenequation deduced from boundary condition at both ends of the sample [CHE 10]. In the case of a weak damping material, at resonance frequency, amplitude of [2.8] is written as:

$$|E^*| \cong Re\,(E^*) = (48\,\pi^2/\beta^4)(m/b)\,(L/h)^3 f^{\,2} \quad [2.9]$$

where β is assimilated to the real root of the eigenvalue equation of a free-clamped rod. In [2.9] the density in [2.8] is replaced by m/ bhL, m being the sample mass.

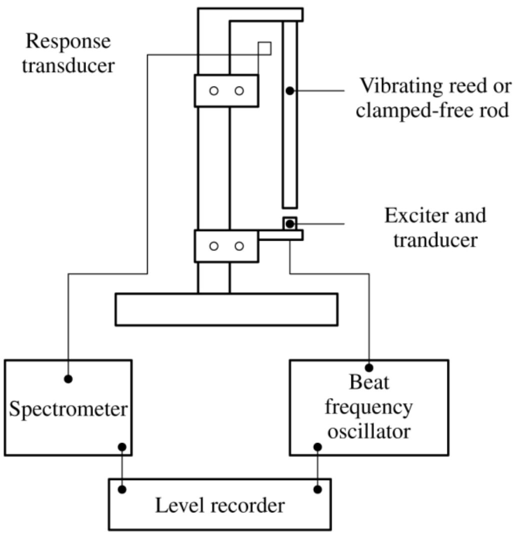

Figure 2.5. *Line diagram of Bruel and Kjaer Oberst apparatus type 3930*

2.3.1. *Comments*

The short length of the sample (7.5 < L < 22 cm) compared to the transverse dimensions (max. 12 x 12 mm) requires caution when using this apparatus if a high resonance frequency is adopted for excitation.

Dispersion of the phase velocity of the wave in the sample must be taken into account, and formulae presented in [CHE 10] (based on Bernoulli–Euler's equation) might be inappropriate. Timoshenko's equation should be used to obtain an appropriate value of eigenvalue β. Globally, at higher frequency, the inertia effect and shear effect cannot be neglected.

Clamping at one end is used for evaluation of the complex Young's modulus and length correction is necessary. This is due to the rod clamping itself when slenderness (defined as the ratio L (length)/h (width)) is not sufficient. Length correction takes into account clamping force; see Chapter 1. Remember that length intervenes as the third power L^3 (see equation [2.9]).

At high temperature, ferromagnetic transducers are used in excitation as well as in measuring displacement. As the instrument is presented to eventually measure the Young's modulus at high temperature (up to 250°C), the ferromagnetic property of the transducers is temperature dependent. Curie's temperature, around 100°C (see Chapter 4), should also be taken into account.

In this apparatus, damping measurements strongly depend on the ferromagnetic force acting on the sample. This force is not negligible, particularly when one measures low damping:

$$10^{-3} < \tan \delta < 10^{-2}$$

Parasitic damping due to electromagnetic force might be of the same order as or higher than the sample damping. Consequently, experimental results might be false.

2.4. Dynamic mechanical analyzer DMA–Dupont de Nemours 980

To our knowledge, this analyzer is the most sophisticated instrument. Our attention is focused on the mechanical conception of the sample holder and measurement system.

2.4.1. *Sample holder*

This holder is composed of a set of arms mounted on low friction pivots whose resonant frequency does not exceed 3 Hz. One of the arms serves to drive the sample while the other one is the counterweight arm and acts as an axis of physical support. This assembly is set to behave like a compound resonance system (sample and arms) driven by an electromechanical transducer to maintain constant flexural amplitude of vibration of the sample at its natural resonant frequency.

The active arm rotates around its pivot and creates a weak deflection of the sample (10^{-1} mm), submitting the sample to bending stress. When the applied bending force is released, the potential energy stored gives rise to free vibration at resonance frequency.

2.4.2. *Measuring the Young's modulus through resonance frequency f_0*

The proposed formula for the Young's modulus is:

$$E = \{[4\pi^2 f_0^2 J_0 - K] / [2H\,(D/2 + L_v)^2]\}.\,(D/B)^3 \qquad [2.10]$$

where J_0 is the inertia moment of the arm, L_v is the damping distance, H the width, B the sample thickness, and D the sample length[1].

Damping measurement is related to energy dissipation of the sample and is directly measured by the amount of power necessary to maintain constant amplitude.

The dimensions of the sample are:

0.02 < Thickness < 1.6 mm

0.02 < Width < 13 mm

10 < Length < 25 mm

The available temperature range in the climatic chamber is -150°C < T < 500°C, whilst the frequency range is narrow, ranging from 3.5 to 150 Hz.

Calculations of Young's modulus $|E^*|$ and damping capacity tan δ_E are made by computer.

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1 The notation adopted in this section is that of the constructor and is different to our own standard notation.

2.4.3. *Comments*

In spite of the complete set up proposed by the manufacturer, the weakness of this system resides in the mechanical parts: the exciter, sample holder and connecting arms. In [2.10] the length correction is the length of the sample parts under clamping grips L_V. This correction needs to be revisited. The clamping force is taken into account.

The length of the sample is short and thus length correction due to clamping becomes important. Clamping compression force is an important factor in the determination of correction length.

Equation [2.10] giving the Young's modulus is based on Bernoulli's equation. A wave dispersion (see Chevalier [CHE 10] for example) of wave velocity versus wave number with slenderness as a parameter, would serve to check the validity of the proposed formula.

When evaluating a material damping coefficient, one must take into account the connection between the sample and the holder. The possible friction between some mechanical components during tests might have an influence. Air damping plays an important role in the energy partition for low material damping measurements:

$(\tan \delta \cong 5.10^{-3})$

2.5. Elasticimeter using progressive wave PPM 5

A sonic technique is used to evaluate the time of flight of a wave packet through a sample. A progressive wave is used instead of stationary waves.

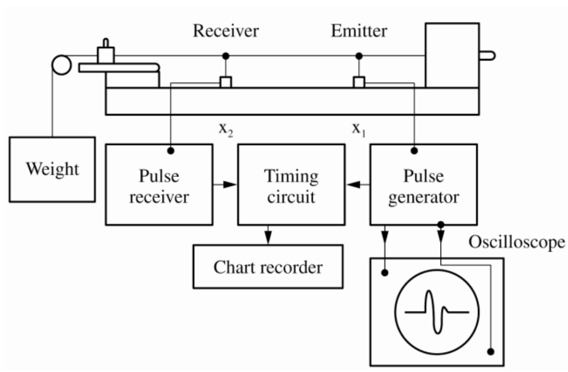

Figure 2.6. *Line diagram of Morgan pulse propagation instrument for string*

From the time of the flight, which depends on the dimensions of rod (or filament), the phase velocity v_p is determined and the longitudinal Young's modulus evaluated, with ρ being the density:

$$E = \rho v_p^2 \tag{2.11}$$

The packet of sinusoids must be short in time so that no parasitic reflecting wave occurs. The PPM 5 (pulse propagation meter) proposed by Henry Morgan is commonly used in the fiber industry. Figure 2.6 shows a diagram of the apparatus.

A complex Young's modulus evaluation is possible by calculating the wave attenuation in space after propagation in the sample (see Figure 2.7).

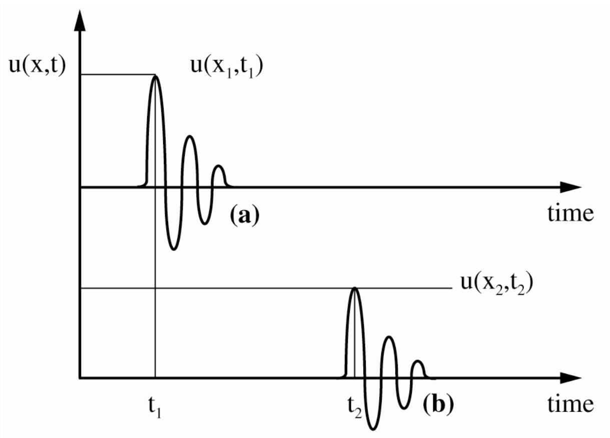

Figure 2.7. *Wave packet propagating along a filament from emitter (x_1) to receiver (x_2) in Figure 2.6. The amplitudes of waves $u(x_1, t_1)$ and $u(x_2, t_2)$ enable wave attenuation to be evaluated from which the damping angle is deduced*

The complex Young's modulus is written as:

$$E^* = |E^*| \exp(j\delta_E) \tag{2.12}$$

The progressive wave is written as follows:

$$u(x,t) = B \exp j(\omega t - k^* x) \tag{2.13}$$

$$k^* = k_1 - jk_2 = \omega [\rho / E^*]^{0.5} \tag{2.14}$$

The complex wave number is related to E^*:

$$k^* = \omega \, [\, \rho / | E^* | \,]^{1/2} \, [\, \cos(\delta_E /2) - j \sin(\delta_E/2)] \quad [2.15]$$

The space wave attenuation is measured by logarithmic decrement:

$$\alpha = [(1/(x_2 - x_1)] \ln [u(x_1 / u(x_2)] \quad [2.16]$$

$$\alpha = k_2 = \omega \, [\rho / | E^* |]^{0.5} \sin(\delta_E / 2) \quad [2.17]$$

From [2.17] it is possible to evaluate the angle $\delta_E/2$. Note in particular the presence of circular frequency ω in equation [2.17]. This is the eigencircular frequency of the emitter if it is a piezoelectric transducer. If not, the circular frequency ω is evaluated via the sinusoids visualized on the oscilloscope screen. It can be also evaluated by using a Fourier transform of the wave packet.

2.6. Bibliography

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