School of Engineering
Institute of Materials
Laboratory of Macromolecular
and Organic Materials

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Course Notes of Chapter 4.2

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1. Introduction: Phenomenological Approach to Viscoelasticity

1.1 What is Viscoelasticity?

At small deformations and at temperatures far from phase transitions, elastic solids (elastomers, glasses, crystals) follow Hooke's law, $\sigma = E\varepsilon$, where the elastic modulus, E, is approximately independent of the time, t, and the strain rate $d\varepsilon/dt$. In contrast, most polymers show a pronounced viscoelastic behavior close to their thermal transitions, i.e., their mechanical properties are strongly influenced by t and $d\varepsilon/dt$ over wide temperature ranges.

The diagram in Slide 231 shows the small deformation behavior of an amorphous polymer over a wide range of temperatures and, compared to previous versions, has been complemented by

School of Engineering
Institute of Materials
Laboratory of Macromolecular
and Organic Materials

Dr. Daniel Görl
daniel.gorl@epfl.ch
lmom.epfl.ch

EPFL STI IMX LMOM
MXG 037, Station 12
CH-1015 Lausanne

that of a semi-crystalline polymer and that of an elastomer. In the case of the semi-crystalline polymer, we always observe a drop in modulus around T_g associated with the amorphous regions. The crystalline part remains solid up to the melting temperature, when a further decrease of the modulus occurs. If the melting temperature is higher than the rubbery plateau of a corresponding amorphous polymer, the rubbery regime can no longer be established. The existence of a rubbery plateau in semi-crystalline polymers requires therefore a fairly high molar mass. In the case of an elastomer, the molar mass is effectively infinite, and the rubbery plateau extends up to the degradation temperature.

We recognize that **the behavior of polymers around transitions such as the glass transition or the edge of the rubbery plateau is strongly dependent on temperature and time**. Indeed, **we observe a qualitatively similar behavior when the temperature increases for a fixed measurement speed, or the time increases (or the frequency decreases) at a constant temperature**. This phenomenon is called **"time-temperature equivalence".** So, if we replaced the T axis of the diagram on Slide 231 by an $\ln t$ axis, the curves remain qualitatively similar, although it is impossible to cover a sufficiently large time range to reproduce the latter behavior in the laboratory during creep measurements. **We wonder later whether it is possible to exploit the time-temperature equivalence quantitatively in order to predict the long-term behavior of a polymer**. We find an analogy in dynamic measurements (Section 2.3), except that an increase in frequency is equivalent to a decrease in effective measurement time and the curves are therefore reversed (Slide 260).

1.2 Importance of Viscoelasticity

We must therefore take the effect of time or frequency in practical applications of polymers into account, as well as that these effects are very sensitive to temperature. The examples given on Slides 232 and 233 are illustrative but not exclusive. An engineer is obviously concerned by **long-term properties** (creep, fatigue, etc.): parts intended for construction, for example, often have to last for more than thirty years. Properties involving **speed** or **very high frequencies** are also very important for applications comprising vibrations, alternating electric fields, or shock absorption. In addition, **energy dissipation** associated with viscoelastic materials can lead to significant overheating and failure of an article. Finally, **viscoelasticity is essential for polymer processing** such as fiber spinning or blow molding of plastic bags.

1.3 Phenomenological Approach to Viscoelasticity

This week we will focus on simple mechanical models based on linear viscoelasticity and different linear combinations of elastic springs and dashpots (dampers) that follow Hooke's and Newton's law, respectively. They are **phenomenological models trying to reproduce the observed phenomena rather than to interpret them in terms of the fundamental physics of the system** (a polymer is obviously not composed of springs and shock absorbers). Phenomenological approaches to nonlinear viscoelasticity will be briefly mentioned but not dealt with explicitly, and we will discuss the molecular approach towards viscoelasticity next week.

2. Linear Viscoelasticity

2.1 Basic Viscoelastic Functions

Slide 237 qualitatively shows the influence of time on stress and strain for different type of tests (creep at constant stress, relaxation at constant strain, simple tension with constant strain rate). "Static" tests such as relaxation and creep allow us to define **viscoelastic functions** for the two main modes of stress, tension and shear:

$$\text{relaxation under tension: } \mathrm{E}(t) = \frac{\sigma(t)}{\varepsilon_0} \qquad \text{relaxation under shear: } \mathrm{G}(t) = \frac{\sigma(t)}{\gamma_0}$$

$$\text{creep under tension: } \mathrm{D}(t) = \frac{\varepsilon(t)}{\sigma_0} \qquad \text{creep under shear: } \mathrm{J}(t) = \frac{\gamma(t)}{\sigma_0} \qquad (1)$$

$\mathrm{E}(t)$ and $\mathrm{G}(t)$ are *moduli* and $\mathrm{D}(t)$ and $\mathrm{J}(t)$ are *compliances*. Those are viscoelastic functions and they are written in straight characters to not confuse them with Young's modulus, E, etc., which does not depend on t. (For a Hookean solid, $E = 1/D$, but, in general, $\mathrm{E}(t) \neq 1/\mathrm{D}(t)$). **If the viscoelastic functions are independent of the stress and deformations, one speaks of linear viscoelasticity**.

2.2 Principle of the Boltzmann Superposition

The deformation or stress of a viscoelastic material following an arbitrary loading history is not defined by an instantaneous stress or strain, unlike a linear elastic solid, where it is enough to know the stress to calculate the strain (or vice versa).

In a linear viscoelastic material, according to the principle of superposition of Boltzmann, **the state of deformation or stress depends on the history of all the stresses applied to the material.** Each new load (strain) applied independently contributes to the final strain (stress). So, if we know the dependence of the modulus or the compliance with t, we can hope to calculate the stress or the strain for a history of deformation or arbitrary stresses. The material keeps a “memory” of all stresses (or deformations) it has undergone.

To illustrate how this works, we will consider the case of tensile creep experiment, where we apply a stress and look at the evolution of the strain. However, instead of keeping the stress constant throughout the test, we will impose a stress $\Delta\sigma_0$ at $t = 0$, $\Delta\sigma_1$ at $t = t_1$, $\Delta\sigma_2$ at $t = t_2$ and so on (Slide 238). According to Equation 1, the strain for a constant $\Delta\sigma$ stress applied to $t = 0$ is

$$\varepsilon(t) = \mathrm{D}(t)\Delta\sigma$$

Thus, according to Boltzmann's superposition principle, at a time $t > t_2$:

$$\varepsilon(t) = \mathrm{D}(t)\Delta\sigma_0 + \mathrm{D}(t-t_1)\Delta\sigma_1 + \mathrm{D}(t-t_2)\Delta\sigma_2 \ldots$$

This is valid for any function $\mathrm{D}(t)$ which is independent of σ and ε, so that in general

$$\varepsilon(t) = \sum_{n=0} \mathrm{D}(t-t_n)\Delta\sigma_n \qquad (2).$$

In the continuous limit, we replace $\Delta\sigma_n$ by $d\sigma$, and we obtain

$$\varepsilon(t) = \int_{-\infty}^{t} \mathrm{D}(t-\xi)\frac{\Delta\sigma}{d\xi}\mathrm{d}\xi \qquad (3),$$

where we use $-\infty$ instead of 0 to take all the mechanical history that the sample would have experienced since its inception into account. That said, in practice, we take 0 as the lower limit, and admit that we are dealing with a "blank" sample at the start of the experiment.

For the same reason, we obtain for a tensile experiment, where we vary ε (relaxation),

$$\sigma(t) = \int_{-\infty}^{t} \mathrm{E}(t-\xi)\frac{\Delta\varepsilon}{d\xi}\mathrm{d}\xi \qquad (4).$$

We will see the usefulness of these formulas in section 2.4.

2.3 Dynamic Measurement Techniques

In **dynamic mechanical analysis** (DMA), a sample is subjected to periodic stress or deformation, generally of sinusoidal form, with an angular frequency ω [rad/s]. The measurement configuration depends on the nature of the material. For example, deformation under tension and bending can be conveniently applied to rather solid materials, while a shear between two plates is often used for rather liquid substances (we are then talking about "rheological" measurements rather than DMA, but this distinction is arbitrary).

The possibility of carrying out the measurements by controlling the stress or the deformation depends on the instrument. In controlled strain,

tensile: $\varepsilon(t) = \varepsilon_0 \sin(\omega t)$ shear: $\gamma(t) = \gamma_0 \sin(\omega t)$ (5),

and the stress is typically measured as a function of the temperature T (constant ω) or as function of ω (constant T). The stress (in shear, τ is sometimes used for the stress) is given by

$$\sigma(t) = \sigma_0 \sin[\omega t + \delta] \qquad (6).$$

Dr. Daniel Görl
daniel.gorl@epfl.ch
lmom.epfl.ch
EPFL STI IMX LMOM
MXG 037, Station 12
CH-1015 Lausanne

There is therefore a phase shift, δ, between the stress and the strain (Slide 243).

The dynamic modulus and the loss factor

We can define the "**dynamic moduli**", as well as the "**loss factor**" in the following manner (here, for a shear test):

$$\sigma(t) = \sigma_0 \sin[\omega t + \delta] = \sigma_0(\sin \omega t \cos \delta + \cos \omega t \sin \delta)$$

$$\sigma(t) = \mathrm{G}' \sin \omega t + \mathrm{G}'' \cos \omega t$$

$$\left.\begin{aligned} \mathrm{G}' &= \frac{\sigma_0}{\gamma_0} \cos \delta \\ \mathrm{G}'' &= \frac{\sigma_0}{\gamma_0} \sin \delta \\ \frac{\mathrm{G}''}{\mathrm{G}'} &= \tan \delta \end{aligned}\right\} \quad (7),$$

where **G' and G'' are respectively the "storage modulus" and the "loss modulus"** (in tension, we replace G by E and γ by ε). These quantities, which typically depend on the frequency of the measurement, can be determined by DMA. **G' reflects the elastic nature of the system, i.e. part of the stress that is in phase with the strain**, and **G'' reflects the viscous character of the system, that is the part of the stress which is out-of-phase with the strain. Thus, tan δ is called the loss or damping factor or coefficient, because the larger it is, i.e. the higher G'' in relation to G', the more dissipative the system.**[1]

Indeed, **in a Hookean solid, σ is equal to $G\gamma$ and therefore G'' = 0**. On the other hand, in a Newtonian liquid σ is equal to $\eta \mathrm{d}\gamma/\mathrm{d}t$, or $\eta\omega\gamma_0 \cos \omega t$ in our example. **So, G' = 0 and G'' = $\eta\omega$.** This is why in rheology, where we are interested in the behavior of liquids, we tend to replace G' and G'' with $\omega\eta''$ and $\omega\eta'$ in Equation 7, where η'' and η' are also viscoelastic functions which depend on ω.

Complex Representation

It is in general easier to get rid of trigonometric functions in calculations by working in the complex domain (by convention we will henceforth use cosines for the deformation).

$$\gamma(t) = \gamma_0 \cos(\omega t) = \Re\gamma^*(t) = \Re\gamma_0 e^{i\omega t}$$

$$\sigma(t) = \sigma_0 \cos(\omega t + \delta) = \Re\sigma^*(t) = \Re\sigma_0 e^{i(\omega t + \delta)} \quad (8).$$

[1] When you deform a viscous liquid, the thermodynamic state of the liquid does not change during deformation - deformation is irreversible and therefore work is dissipated as heat.

Dr. Daniel Görl
daniel.gorl@epfl.ch
lmom.epfl.ch

EPFL STI IMX LMOM
MXG 037, Station 12
CH-1015 Lausanne

We can then define a "**complex modulus**", G*, such that

$$\mathrm{G}^* = \frac{\sigma^*(t)}{\gamma^*(t)} = \frac{\sigma_0 e^{i(\omega t+\delta)}}{\gamma_0 e^{i\omega t}} = \frac{\sigma_0 e^{i\delta}}{\gamma_0} = \frac{\sigma_0 \cos\delta + \sigma_0 i \sin\delta}{\gamma_0} = \mathrm{G}' + i\mathrm{G}'' \qquad (9).$$

You will see the usefulness of this approach later.

2.4 Simple Phenomenological Models

We saw in Section 2.3 that the linear viscoelastic behavior is intermediate between that of a Newtonian liquid and that of a Hookean solid. **In order to model the viscoelastic behavior of polymer materials, we can try out linear combinations of these two types of behavior, based on "springs" and "dashpots" ("shock absorber")** (Slide 247).

Maxwell in Simple Tension / Relaxation

Maxwell's model (Slides 249-252) consists of a Hookean spring of (constant) modulus E and a Newtonian dashpot of viscosity η that are connected in a series to model a deformation in tension. This is the simplest model to combine a spring and a dashpot, but nevertheless an informative model and, as we will see later, quite realistic in some cases. So, let's apply a constant deformation ε_0 at $t = 0$. As the two elements are in series, the same stress σ is applied to both:

$$\varepsilon_0 = \varepsilon_{spring}(t) + \varepsilon_{dashpot}(t) \quad \Rightarrow 0 = \frac{d\varepsilon_{spring}}{dt} + \frac{d\varepsilon_{dashpot}}{dt}$$

$$\frac{1}{E}\frac{d\sigma}{dt} + \frac{\sigma}{\eta} = 0$$

$$\frac{d\sigma}{\sigma} = -\frac{E}{\eta}dt \Longrightarrow {}_{\sigma_0}^{\sigma}[\ln\sigma] = -\frac{E}{\eta}t \Longrightarrow \sigma = \sigma_0 e^{-\frac{E}{\eta}t} \equiv \sigma_0 e^{-\frac{t}{\tau}}$$

where $\boldsymbol{\tau = \eta/E}$ **is the "relaxation time"**. So, the relaxation modulus

$$\mathrm{E}(t) = \frac{\sigma(t)}{\varepsilon_0} = Ee^{-\frac{t}{\tau}} \qquad (10).$$

This exponential decrease of the stress (Slide 249) is at least *qualitatively* similar to what you see when you do a relaxation test on a polymer near T_g.

Maxwell in Dynamic Tension

We will now try to predict the behavior according to the angular frequency ω of the Maxwell's model for sinusoidal deformation. For this we need the Equation 4:

$$\sigma(t) = \int_{-\infty}^{t} \mathrm{E}(t-\xi)\frac{\Delta\varepsilon}{d\xi}\mathrm{d}\xi$$

as well as the viscoelastic law (Equation 10) that we have just established. The complex deformation is now

$$\varepsilon^* = \varepsilon_0 e^{i\omega t} \Rightarrow \frac{d\varepsilon^*}{dt} = i\omega\varepsilon_0 e^{i\omega t}$$

So:

$$\sigma^* = \int_{-\infty}^{t} Ei\omega\varepsilon_0 e^{-\frac{t-\xi}{\tau}} e^{i\omega\xi} d\xi$$

$$= \left[\frac{Ei\omega\varepsilon_0 e^{-\left(\frac{t-\xi}{\tau}-i\omega\xi\right)}}{\frac{1}{\tau}+i\omega}\right]_{-\infty}^{t} = \frac{Ei\omega\varepsilon_0 e^{i\omega t}}{\frac{1}{\tau}+i\omega} = \frac{\left(\frac{1}{\tau}-i\omega\right)Ei\omega\varepsilon_0 e^{i\omega t}}{\left(\frac{1}{\tau}-i\omega\right)\left(\frac{1}{\tau}+i\omega\right)} = \frac{\left(\frac{1}{\tau}-i\omega\right)Ei\omega\varepsilon_0 e^{i\omega}}{\frac{1}{\tau^2}+\omega^2}$$

$$\mathrm{E}^* = \frac{\sigma^*}{\varepsilon^*} = E\frac{i\omega\left(\frac{1}{\tau}-i\omega\right)}{\frac{1}{\tau^2}+\omega^2} = \mathrm{E}' + i\mathrm{E}'' = E\frac{\tau^2\omega^2}{1+\tau^2\omega^2} + iE\frac{\omega\tau}{1+\tau^2\omega^2} \qquad (11).$$

We therefore found our storage modulus and loss modulus, which strongly depend on the frequency of the deformation. We notice, for example, that in the limit where $\omega \ll 1/\tau$, the so-called "**terminal regime**", E' is proportional to ω^2 and E" is proportional to ω (Slides 251).

As shown in Slide 252, we can roughly observe this behavior for simple systems which are characterized by a single relaxation time, a constant storage modulus at the highest frequencies, a transition at $\omega \ll 1/\tau$ marked by a peak of E'' or G'' and therefore also of tan δ, and a terminal regime. However, this remains too simple for polymers (Slide 253), where we observe, for example, a rubbery plateau at low frequencies (which is equivalent to the temperature range of the rubbery state according to the time-temperature superposition principle).

Zener in dynamic tension

In the **Zener model** (also known as the "**standard linear solid**") an E_B spring is put in series with **the Voigt model** containing an E_A spring (Slide 255). After some calculations, we find:

$$\mathrm{E}'(\omega) = E_B - \frac{E_B - E_\infty}{1+\tau_\delta^2\omega^2}, \quad \mathrm{E}''(\omega) = \tau_\delta\omega\frac{E_B - E_\infty}{1+\tau_\delta^2\omega^2},$$

$$E_\infty = \frac{E_A E_B}{E_A + E_B}, \quad \tau_\delta = \frac{\eta(E_B - E_\infty)}{E_B^2} \qquad (12).$$

Dr. Daniel Görl
daniel.gorl@epfl.ch
lmom.epfl.ch

EPFL STI IMX LMOM
MXG 037, Station 12
CH-1015 Lausanne

Indeed, with three elements we can better simulate the rheological behavior of a real polymer (Slide 256). E” always shows a peak at the glass transition, but E’ tends towards E_∞ when $\omega \ll 1/\tau_\delta$, and we find the “rubbery plateau” of the polymer. Note that if E_A = 0, the storage modulus becomes that of the Maxwell model:

$$\mathrm{E}'(\omega) = E_B - \frac{E_B}{1+\tau^2\omega^2} = \frac{\tau^2\omega^2 E_B}{1+\tau^2\omega^2}$$

Generalized models

Instead of modelling the viscoelastic behavior of a polymer with Equation 12, **we can do better by adding additional Voigt elements. Thus, in the generalized Voigt-Maxwell model (Slide 257), we accumulate an arbitrary number of elements, and the behavior is described by a series** of shape

$$\mathrm{E}(t) = E_\infty + \sum_1^n E_i e^{-\frac{t}{\tau_i}} \qquad (13).$$

And for the dynamic moduli

$$\begin{aligned} \mathrm{E}'(\omega) &= E_\infty + \sum_1^n E_i \frac{\tau^2\omega^2}{1+\tau^2\omega^2} \\ \mathrm{E}''(\omega) &= E_\infty + \sum_1^n E_i \frac{\tau\omega}{1+\tau^2\omega^2} \end{aligned} \qquad (14).$$

We can also use a continuous expression:

$$\mathrm{E}(t) = E_\infty + \int_0^\infty \mathrm{H}(\tau)\, e^{-\frac{t}{\tau}}\, d\ln\tau \qquad (15).$$

where $\mathrm{H}(\tau)$ is a **relaxation time spectrum.** Similar expressions exist for the other viscoelastic functions as well as for the dynamic moduli. It should be emphasized that **these are purely empirical expressions.** Moreover, their use is only justified if the same model describes the entire viscoelastic behavior (relaxation, creep, dynamic stress), which is not always verified experimentally (it is assumed that the relaxation time spectrum changes during the experiment in these cases, for example, if we vary the temperature).

We also know relatively simple empirical expressions like the Cole-Cole Equation (Slide 257), which have the advantage to rely on fewer parameters, but this does not allow us to get closer to a physical interpretation of the observed phenomena.

2.5 Nonlinear Viscoelasticity

As we have seen, **at large deformations, which are of major importance for practical applications, the moduli and compliances of polymers begin to depend on the stress and strain and the Boltzmann superposition becomes inapplicable**. The phenomenological models of nonlinear viscoelasticity are often extensions of the (mostly empirical) linear models presented here. They are certainly useful to an engineer but their interpretation is difficult. This is why the experimental characterization techniques (DMA) are typically applied to the linear viscoelastic regime.

3. Time-Temperature Equivalence

3.1 Time-Temperature Superposition

The principle of time-temperature equivalence is well established by experimental observations, but it is still necessary to quantitatively link the time- or frequency-dependent behavior to the evolution of viscoelastic functions with temperature. With the help of short-term tests at different temperatures, this will make it possible to reconstruct the behavior over very wide ranges of frequency or time that are inaccessible in the laboratory.

Already empirically, we find that **data for E'(ω) or E''(ω) (for example, obtained at different temperatures above T_g, and corrected by a factor ρT which comes from the expression for the modulus of an elastomer $E = 3NkT$ and which is often negligible if the variations of E are very large) can be superimposed with a curve obtained at a reference temperature, T_r, by multiplying ω with a displacement factor a_T (T, T_r)**. We thus obtain a "master curve" for the behavior at T_r which covers a very wide range of frequencies.

3.2 The Williams-Landel-Ferry (WLF) Equation

The displacement factors ω_r/ω follow the empirical Williams-Landel-Ferry (WLF) equation:

$$\log a_T = -\frac{C_1(T - T_r)}{C_2 + T - T_r} \qquad (16).$$

where C_1 and C_2 are empirical constants. Moreover, if we take T_g as the reference temperature, we **obtain "universal" values for C_1 and C_2, namely 17.44 and 51.6 K** respectively, which more or less describe the behavior of any amorphous polymer.

Is it possible to justify this approach? We have already seen expressions for the temperature-dependence of a relaxation time in the context of the free volume theory (Chapter 4.3) for the glass transition:

$$\tau(T) = \tau_0 e^{\frac{v_0}{v_{fm}}} = \tau_0 e^{\frac{\Delta\alpha^{-1}}{T-T_0}} \qquad (17).$$

where T_0 is a characteristic temperature of the material and $\Delta\alpha$ is the difference between the thermal expansion coefficients below and above T_g. Let $T_0 = T_g - A$:

$$\tau(T) = \tau_0 e^{\frac{\Delta\alpha^{-1}}{A+T-T_g}}, \qquad \tau(T_g) = \tau_0 e^{\frac{\Delta\alpha^{-1}}{A}} \tag{18}$$

In general, the same behavior is expected at different T for equal values of $\tau(T)\omega$, where $\tau(T)$ is the time needed to activate a mechanism and ω^{-1} is the time available. Thus $a_T = \omega_{Tg}/\omega = \tau(T)/\tau(T_g)$ and

$$\ln a_T = \ln \tau(T) - \ln \tau(T_g) = \frac{\Delta\alpha^{-1}}{A+T-T_g} - \frac{\Delta\alpha^{-1}}{A}$$

$$\ln a_T = \frac{A^{-1}\Delta\alpha^{-1}(T-T_g)}{A+T-T_g} \Rightarrow \log a_T = -\frac{C_1(T-T_r)}{C_2+T-T_r} \tag{19}$$

This remains valid if the transition is characterized by a relaxation time spectrum with all relaxation times having the same dependence on T. Even though this condition is met, the "universal" constants of Equation 16 are not always very precise, and it is necessary to determine them empirically for each material.

3.3 General Comments on the WLF Approach

As we have seen, the WLF approach is very useful because the experimentally accessible range of frequencies or time is limited and the displacement factors make it possible to extend this range using measurements taken at other temperatures to construct a master curve. We can therefore predict the creep behavior, for example, at extremely long times.

However, even assuming that the theory is correct, its application is limited to $T_g < T < T_g + 50$ K (approximately), i.e. the temperature range where the behavior is strongly influenced by the glass transition. If there is too much overlap with other processes whose relaxation times do not have the same dependence on temperature as those of the glass transition, the superposition becomes approximate if not impossible. Moreover, this approach cannot be applied to the nonlinear viscoelastic behavior in case of large deformations. Finally, it can only be justified if all viscoelastic functions superimpose.

4. Summary

- Polymers are viscoelastic materials, their mechanical behavior is highly sensitive to the timescale of the measurement.
- Linear viscoelasticity may be applied when the deformations are small, allowing the application of simple models for this time-dependent behavior.

• Time-temperature equivalence is a qualitative feature of the behavior of polymeric materials. It is invoked to justify the practice of time-temperature superposition, which can be used to greatly extend the time or frequency range of measurements.

Dr. Daniel Görl
daniel.gorl@epfl.ch
lmom.epfl.ch

EPFL STI IMX LMOM
MXG 037, Station 12
CH-1015 Lausanne