RESEARCH ARTICLE

DYNAMIC THREE-POINTS–BENDING TEST MODE OF TWO AMORPHOUS POLYMER MATERIALS (PMMA, PC) FOR THEIR VISCOELASTIC AND MECHANICAL CHARACTERIZATION

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Abstract
This paper presents an approach to classifying amorphous polymer materials. Temperature is this classification involves the determination of mechanical and viscoelastic characteristics considered a descriptive variable to clarify the specific field of practical applications of amorphous polymers, according to the reference temperature characterizing the behavior of polymer materials. The mechanical and viscoelastic characteristics of amorphous polymers such as methyl poly-methacrylate (PMMA), polycarbonate (PC) and imide poly ether (PEI) are determined through the three-point dynamically embedded test carried out in an adiabatic close enclosure. The complex dissipative or conservative modules according to the temperature are represented. The results obtained show that the fluidity index of these materials is linked to their viscosity, which is a determining property which is decisive for the choice of the technique of the application of the material. Our method of measuring properties therefore, in principle, comparable to the techniques used in industrial development.

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Introduction:
By considering temperature as a descriptive variable for the classification of polymer materials, the specific area of their specific properties can be specified in terms of the viscoelastic characteristics of solid state. To these properties, it is possible to associate the notion of transition from the rigid solid state to the rubberized state, in terms of their flow, which is more characteristic of their viscous liquid state.

It should be noted that passages such as: pasty solid–solid, viscous rigid–solid, viscous liquid–solid, solid–liquid, liquid–liquid, are sometimes difficult to understand. In fact, these changes in the condition of materials derived from polymers are sometimes dependent, in addition to the tested temperature, the mode of solicitation and the time of application. The reference temperatures that characterize the behavior changes of polymer materials, thanks to these thermal experimental benchmarks, are mainly [1–12]:
1. the freezing temperature,
2. the glass transition temperature,
3. the temperature of fragilization,
4. the melting temperature for polymers,
5. melting temperature in the case of semi-crystalline polymers,
6. and the Vicat softening temperature, similar to the under-load softening temperature.

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For this reason, it has seemed important to carry out in an adiabatic closed chamber, different three–points–bending solicitations of the selected polymers: amorphous polymers such as poly(methyl methacrylate) (PMMA), polycarbonate (PC), Polyetherimide (PEI), for their mechanical viscoelastic characterization. These different experiments on these materials in dynamic mode using the three–points–bending tests with a viscoelastic meter, to determine the dynamic quantities in terms of complex modulus, loss factor in order to achieve a classification of the observed behaviours. The resulting representations are of the following type: modulus (dissipative or conservative) according to temperature f(T). This will help identify their practical application areas.

The benefit of this approach is that general laws of rheological behavior can be effectively uncovered without involving molecular approaches to these descriptive approaches.

**Equipment**

Equipment which can in be considered as a capillary rheometer, is a viscoelastic-meter which has been used to impose different loads on the material in order to obtain experimental data that represent the evolution of the stresses according to the rate of deformation such as:

$$\tau = f(\gamma)$$

(1)

**It is on this basis that the work carried out was implemented with particular attention:**

First, the experimental determination of typical temperatures and this follows different complementary approaches. The variability or conservativeness of these data is associated with the different selected materials classified in the first approach according to the value of their fluidity index.

1. Second, to the rheological changes in polymers taken as a model of behaviour depending on different types of apparatus commonly used or not for these determinations.
2. The originality of the approach chosen concerns different geometric configurations, sometimes out-of-standard, retained and their extrapolation allowing to obtain descriptive rheograms of the properties of use.

**Method:**

In the three–points–bending test (Fig. 1), the sample is anchored in the middle by two clamps connected to the viscoanalyzer’s movable base determining the strain signal. The mounting of the sample is ensured by two ends attached to the base of mechanical structure of the equipment. The whole is enclosed in a stated thermal enclosure. The sample support and the dynamic excitation system are designed to work without pre–stress in order to avoid the creep of the material.

![Figure 1: Schematic representation of the three–points–bending test of the sample](image)

The end of the sample is encased by two mors attached to the base of the mechanical structure of the apparatus. In order to remain in pure bending, the sample must be sized so that the length ratio between the support L normalized to the thickness h is greater than the value of 15 recommended by the manufacturer. In addition, resonance effects at the support level limit measurements to frequencies below 200 Hz. Support efforts (moments and forces) at the various enclosures are easily determinable [12, 13]. For example, [13, 14] it is easy to find the maximum arrow that the sample receives when it is solicited from the expression of the \(y_{\text{max}}\) deflection (Eq. (2)):

$$\frac{F \cdot L^3}{16E \cdot b \cdot h^3} = y_{\text{max}}$$

(2)
Where F is the force and L, h, b, are dimensions of the sample. The force F, linked to the excitation signal x, is known via the dynamic stiffness module K*, which is obtained by the force sensor released by the apparatus. This force is written (Eq. (3):
\[ F \square k^* \square x(t) \]  (3)

The complex dynamic flexion module is then inferred from this:
\[ I. \quad E L^3 \quad K L^3 \quad y_{max} \]  (4)

The y_{max} deflection is the out-of-phase sinusoidal response of d associated with the excitation signal x and their ratio gives us the off set factors. Hence the components E',
\[ E' = \frac{K L^3}{16 b h^3} \]
\[ E'' = \frac{15.6 L^3}{64 b h^3} \]  (5)

The second term of E' is a correction factor due to the experimental conditions and intrinsic properties of the material.

**Results and Discussion:**

The samples of tested solid polymers are cut into plates of thickness between 3 and 5 mm and then mounted embedded according to the diagram in figure 1.

The viscoelastic features in three point embedded flexion were determined using Eqs. (5) to (7). In the case of PMMA, there is no significant difference between the three-point bending tests (figures 2 to 4).

Descriptive quantities of the value of the dynamic glass transition appear almost conservative regardless of the selected mode.

The same behaviour, slightly equivalent on E' = f(T ) values, but stills lightly higher amplitudes of E'' = f(T ) sizes for three-points bending tests, are found in the polycarbonate tests (figures 2 to 5 on the one hand and figures 5 to 7 on the other).

Figures 2 and 5 confirm the respective behaviour of PMMA and PC polymers as shown by Rinaldi’s thesis work [15]. As for figures 3, 4, 6 and 7, it is noted that the peaks observed on the curves correspond effectively to the glass transition temperatures of the two selected materials. This reflects the relaxing aspect [16] of these materials.

A slight difference in scale and shape is also observed depending on the nature of the test selected and, in particular, a three-points bending modulus E' is higher than the modulus E' in other soliciting modes such as compression-traction [12].

The glass transition temperature of polycarbonate is the same for this type of test (figures 5 and 7).
Figure 2: Conservative Module E’ of PMMA in the three-point embedded flexion.

Figure 3: Dissipative Module E” of PMMA in the three-point embedded flexion.

Figure 4: Loss factor tand of PMMA in the three-point embedded flexion.

Figure 5: Conservative Module E’ of PC (Polycarbonate) in the three-point embedded flexion.
Conclusion:
Using the three-point embedded flexion tests, the viscoelastic quantities $E'$, $E''$ and $\tan(\delta)$ were qualified by temperature for samples of polymers differentiated by their fluidity index.

It is noted that stiffness is an intermediate quantity that makes easy to qualify the conservative $E'$ and dissipative $E''$ components of the complex module $E^*$ and its glass transition temperature of the polymer through the loss factor. These quantities, in comparison with the data provided, allow us to make clear by this method of solicitation that:

the glass transition temperature $T_g$ obtained on the two PMMA samples fits into the temperature range [110-135°C] recommended by the manufacturers of this material. However, a variation of 5°C was observed on the values of this parameter.

the grades of the polycarbonate tested showed a behaviour consistency with a $T_g$ nearly equivalent to that described by the manufacturer’s data.

It should be noted that the values of the softening temperature of these polymers are slightly higher or close to those associated with Heat Deflection Temperature (HDT) and Vicat temperatures. In the end, it is permissible to consider that the fluidity index of the viscoelastic material is linked to its viscosity, a property which is decisive for the choice of the technique of application of the material.

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