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Plastics — Determination of dynamic mechanical properties —

Part 6: Shear vibration — Non-resonance method

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 6721-6:1996), which has been technically revised. It also incorporates the Amendment ISO 6721-6:1996/Amd.1:2007. The main changes compared to the previous edition are as follows:

— the document has been revised editorially;

— normative references have been changed to undated.

A list of all parts in the ISO 6721 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Plastics — Determination of dynamic mechanical properties —

Part 6: Shear vibration — Non-resonance method

1 Scope

This document describes a forced, non-resonance method for determining the components of the shear complex modulus G^* of polymers at frequencies typically in the range 0,01 Hz to 100 Hz. Higher-frequency measurements can be made, but significant errors in the dynamic properties measured are likely to result (see 10.2.2 and 10.2.3). The method is suitable for measuring dynamic storage moduli in the range 0,1 MPa to 50 MPa.

NOTE Although materials with moduli greater than 50 MPa can be studied, more accurate measurements of their dynamic shear properties can be made using a torsional mode of deformation (see ISO 6721-2 and ISO 6721-7).

This method is particularly suited to the measurement of loss factors greater than 0,02 and can therefore be conveniently used to study the variation of dynamic properties with temperature and frequency through most of the glass-rubber relaxation region (see ISO 6721-1). The availability of data determined over wide ranges of both frequency and temperature enables master plots to be derived, using frequency/temperature shift procedures, which display dynamic properties over an extended frequency range at different temperatures.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6721-1, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6721-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

A test-specimen assembly is subjected to a sinusoidal shear force or deformation at a frequency significantly below the fundamental shear resonance frequency (see 10.2.2). The amplitudes of the force and displacement cycles applied to the test-specimen assembly and the phase angle between these cycles are measured. The storage and loss components of the shear complex modulus and the loss factor are calculated using formulae given in Clause 10.

5 Apparatus

5.1 Loading assembly

5.1.1 General

The requirements for the loading assembly are that it shall permit measurements of the amplitudes of, and phase angle between, the force and displacement cycles for a test specimen assembly subjected to a sinusoidal shear force or deformation. Various designs of apparatus are possible: a suitable version is shown schematically in Figure 1. The shear test-specimen assembly consists of two identical specimens S of the polymer bonded to or clamped between metal end-pieces P_1 and P_2. A sinusoidal force is generated by the vibrator V and applied to the two outer end-pieces P_1 of the test-specimen assembly through the clamping device C_1 of the shear load stage. The amplitude and frequency of the vibrator table displacement are variable and monitored by the transducer D. The test-specimen assembly is held at its centre P_2 by a fixed clamp C_2, and thus each specimen S of the polymer is subjected to simple shear deformations of equal magnitude. The sinusoidal force applied in deforming the test-specimen assembly is monitored by a force transducer F connected to C_2.

Alternatively, the force can also be calculated from the current supplied to the vibrator.

The members between the clamps C_1 and V, and between C_2 and F, shall be much stiffer than the test-specimen assembly and shall have a low thermal conductance if the test-specimen assembly is to be enclosed in a temperature-controlled cabinet.

While each member of the loading assembly may have a much higher stiffness than the test-specimen assembly, the presence of clamped or bolted connections can significantly increase the apparatus compliance. It may then be necessary to apply a compliance correction as described in 10.2.4.

A clamping arrangement may be used in which a single specimen of the polymer is subjected to a simple shear deformation, but precautions shall then be taken to ensure that any torque in the loading assembly resulting from the application of load to the specimen does not influence the measurements of the dynamic shear force and displacement. Measurements of the deformation of the specimen may also be made by locating the displacement transducer so as to measure the relative displacement of the two parts C_1 and C_2 of the load stage. The magnitude of the correction for the compliance of the loading assembly will then become small or negligible (see 10.2.4).

5.1.2 Load stage

The shear load stage shall be capable of gripping the test-specimen assembly with sufficient force to prevent any relative movement between the metal blocks P of the test-specimen assembly and the load stage clamps, and to maintain the force at low temperatures. Any misalignment of the load stage with respect to the force transducer will produce a lateral component of the force applied to the transducer during loading of the test-specimen assembly. The alignment of the loading assembly and test-specimen assembly shall be such that any lateral component recorded by the transducer is less than 1 % of the applied longitudinal force.

5.1.3 Transducers

The term transducer in this document refers to any device capable of measuring the applied force or displacement, or the ratio of these quantities, as a function of time. The calibration of the transducers shall be traceable to national standards for the measurement of force and length. The calibration shall be accurate to ± 2 % of the minimum force and displacement cycle amplitudes applied to the test-specimen assembly for the purpose of determining dynamic properties.

5.2 Electronic data-processing equipment

Data-processing equipment shall be capable of recording the force and displacement cycle amplitudes to an accuracy of ± 1 %, the phase angle between the force and displacement cycles to an accuracy of ± 0,1° and the frequency to an accuracy of ± 10 %.

5.3 Temperature measurement and control

According to ISO 6721-1.

5.4 Devices for measuring test specimen dimensions

According to ISO 6721-1.

6 Test specimens

6.1 General

According to ISO 6721-1.

6.2 Shape and dimensions

Various shear test specimen assemblies can be used. A suitable design is shown in Figure 2. Here the metal end-pieces P are cylindrical, but any cross-sectional shape is suitable as long as the end-pieces can be clamped rigidly in the shear load stage. The dimensions of the end-pieces and the polymer specimens S shall be chosen such that the deformation of the end-pieces under an applied load is negligible in comparison with that of the specimens. For a polymer whose shear modulus is less than 100 MPa, this will mean that the thickness of the end-pieces may be comparable with the thickness L of the specimens.

The cross-sectional shape of the polymer specimens in the plane of their bonded faces is not critical, although a rectangular section is recommended in order to simplify the application of a term representing the contribution to the specimen deformation from bending. See Formula (1).

The specimens are typically cut from a sheet of the polymer and bonded to the end-pieces to construct the shear test-specimen assembly. The dimensions of each polymer specimen shall not vary by more than 3 % of the mean value. This dimension shall be sufficiently large to allow adequate accuracy to be achieved in the determination of dynamic strain and hence dynamic moduli [see Formula (1)]. In addition, it is recommended that the dimension h of the polymer in the direction of the applied load should be greater than $4L$ in order to make the correction for bending negligible.

NOTE A variation in dynamic properties can be observed between specimens of different thickness prepared by injection moulding owing to differences which can be present in the structure of the polymer in each specimen.

6.3 Preparation of polymer specimens

According to ISO 6721-1.

7 Number of test specimens

According to ISO 6721-1.

8 Conditioning

According to ISO 6721-1.

9 Procedure

9.1 Test atmosphere

According to ISO 6721-1.

9.2 Measuring the cross-section of the polymer specimen

According to ISO 6721-1.

9.3 Clamping the test assembly

Mount the test specimen assembly in the load stage using a clamping force that is sufficient to prevent relative movement between each clamp and the associated end-piece under all test conditions.

9.4 Varying the temperature

According to ISO 6721-1.

9.5 Performing the test

Apply to the shear test-specimen assembly a dynamic force which yields force and displacement signal amplitudes which can be measured by the transducers to the accuracy specified in 5.1.3.

If the shear strain exceeds the limit for linear behaviour, then the derived dynamic properties will depend on the magnitude of the applied strain. This limit varies with the composition of the polymer and the temperature, and is typically in the region of 0,2 % for glassy plastics, but the effect is evident at very low dynamic strains in carbon-particle-filled rubbers. The dynamic strain range for linear behaviour can be explored by varying the dynamic displacement amplitude at a constant frequency and recording any change in dynamic stiffness with strain amplitude. A low frequency should be used for this purpose to minimize any temperature increase caused by mechanical loss. If nonlinear behaviour is detected in the strain range of interest, the dynamic strain limit should be recorded in the test report.

Record the amplitudes of the phase difference between and the frequencies of the force and displacement signals, as well as the temperature of the test. Where measurements are to be made over ranges of frequency and temperature, it is recommended that the lowest temperature be selected first and measurements made increasing frequency, keeping the temperature constant. The frequency range is then repeated at the next higher temperature (see ISO 6721-1).

For test conditions under which the polymer exhibits medium or high loss (for example in the glass-rubber transition region), the energy dissipated by the polymer may raise its temperature sufficiently to give a significant change in dynamic properties. Any temperature rise will increase rapidly with increasing strain amplitude and frequency. If the data-processing equipement is capable of analysing the transducer outputs within the first few cycles, then the influence of any temperature rise will be minimized. Subsequent measurements will then change with time as the specimen temperature continues to rise, and such observations will indicate the need to exercise some caution in the presentation and interpretation of results.

10 Expression of results

10.1 Symbols

A	bonded area of the specimens, in square metres
f	measurement frequency, in hertz
f_F	resonance frequency of the force transducer, in hertz
f_S	resonance frequency of the test-specimen, in hertz
G'_a, G'	assembly apparent and corrected shear storage modulus, in pascals
G''	shear loss modulus, in pascals
h	mean of the specimen heights, in metres, in the direction of the applied load
k_a, k	measured and corrected magnitude of the complex stiffness of the test-specimen assembly, in newtons per metre
k_F	stiffness of the force transducer, in newtons per metre
k_∞	measured stiffness of a metal bar, in newtons per metre, whose cross-sectional dimensions are the same as those of the end-pieces of the shear test-specimen assembly (see Note). This bar shall be at least 100 times stiffer than the stiffest polymer specimen to be tested
L	mean of dimension of each polymer specimen between bonded faces, in metres
m_F	mass of that part of the loading assembly between the force transducer and the test-specimen assembly, in kilograms
s_A	measured amplitude of the dynamic displacement, in metres
$\tan \delta_{Ga}$, $\tan \delta_G$	apparent and corrected shear loss factor
δ_{Ga}, δ_G	measured and corrected phase difference, in degrees, between the force and displacement cycles
ΔF_A	measured amplitude of the dynamic force, in newtons

NOTE The magnitude of k_∞ will give an estimate of the stiffness of the loading assembly which is equivalent to a spring connected in series with the shear test-specimen assembly and will enable a correction for apparatus compliance to be deduced (see 10.2.4).

10.2 Calculation of the shear storage modulus G'

10.2.1 General

An approximate value for the shear storage modulus G'_a is determined from Formula (1):

$$G'_a = \frac{\Delta F_A}{s_A} \times \frac{L}{A} \times \left[1 + \frac{L^2}{h^2} \times \frac{G'}{E'} \right] \cos \delta_{Ga} = \frac{k_a L}{A} \left[1 + \frac{L^2}{h^2} \times \frac{G'}{E'} \right] \cos \delta_{Ga} \qquad (1)$$

The term in square brackets accounts for a contribution from bending to the deformation of the specimen. Values for G'/E' typically range from 0,37 for isotropic glassy or semicrystalline polymers to 0,33 for rubbers.