
**Plastics — Determination of the transient
extensional viscosity of polymer melts**

*Plastiques — Détermination de la viscosité élongationnelle transitoire
des polymères à l'état fondu*

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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Plastics — Determination of the transient extensional viscosity of polymer melts

1 Scope

This International Standard specifies the general principles of a method for determining the transient extensional viscosity of polymer melts. The procedure details the measurement of polymer melt specimens stretched uniaxially under conditions of constant strain rate and constant temperature.

The method is capable of measuring the transient extensional viscosity of polymer melts at Hencky strain rates typically in the range $0,01 \text{ s}^{-1}$ to 1 s^{-1} , at Hencky strains up to approximately 4 and at temperatures up to approximately $250 \text{ }^{\circ}\text{C}$ (see Notes 1 and 2). It is suitable for measuring transient extensional viscosity values typically in the range from approximately $10^4 \text{ Pa}\cdot\text{s}$ to $10^7 \text{ Pa}\cdot\text{s}$ (see Note 3).

NOTE 1 Hencky strains and strain rates are used (see Clause 3).

NOTE 2 Values of strain, strain rate and temperature outside these limiting values may be attained.

NOTE 3 The operating limit of an instrument, in terms of the lowest transient extensional viscosity values that can be measured, is due to a combination of factors, including the ability of the specimen to maintain its shape during testing and the resolution of the instrument.

A list of documents related to this International Standard is given in the Bibliography.

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2 Normative references

The following referenced documents are indispensable for the application 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 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472, together with the following, apply.

Definitions 3.1 to 3.5 are given by Whorlow^[1] for strains and strain rates, and by the Nomenclature Committee of the Society of Rheology for start-up flow in tensile uniaxial extension at constant Hencky strain rate^[2].

3.1

Hencky strain

ε

strain given by the natural logarithm of the elongation ratio

$$\varepsilon = \ln(l/l_0) \quad (1)$$

where l is the specimen length and l_0 is the original specimen length

NOTE 1 It is also referred to as the natural or true strain.

NOTE 2 It is dimensionless.

**3.2
Hencky strain rate**

$\dot{\epsilon}$
rate of change of Hencky strain with time, given by

$$\dot{\epsilon} = 1/l \times \partial l / \partial t \tag{2}$$

where t is time

NOTE 1 It is independent of the original specimen length l_0 .

NOTE 2 It is expressed in reciprocal seconds.

**3.3
net tensile stress**

σ_E
for tensile uniaxial extension, stress given by

$$\sigma_E = \sigma_{11} - \sigma_{22} = \sigma_{11} - \sigma_{33} = \sigma_{zz} - \sigma_{rr} \tag{3}$$

where σ_{ii} is a stress tensor in either rectangular or axisymmetric co-ordinates

NOTE 1 The tensile stress growth function is indicated by σ_E^+ where the $+$ indicates start-up of flow.

NOTE 2 Net tensile stress is expressed in pascals.

**3.4
tensile stress growth coefficient**

η_E^+
ratio of the net tensile stress to the Hencky strain rate

$$\eta_E^+(t, \dot{\epsilon}) = \sigma_E / \dot{\epsilon} \tag{4}$$

for tensile uniaxial extension, where t is time and $+$ indicates start-up of flow

NOTE 1 It is also known for the purposes of this International Standard as “transient extensional viscosity”.

NOTE 2 It is a transient term.

NOTE 3 It is expressed in pascal seconds.

**3.5
tensile viscosity**

η_E
term given by

$$\eta_E(t, \dot{\epsilon}) = \lim_{t \rightarrow \infty} [\eta_E^+(t, \dot{\epsilon})] \tag{5}$$

NOTE 1 It is the limiting tensile stress growth coefficient value and represents an equilibrium extensional viscosity if a steady value is achieved. However, for materials that do not exhibit a steady-state behaviour, the use of an “equilibrium extensional viscosity” such as this is not appropriate.

NOTE 2 It is expressed in pascal seconds.

4 General principles

In contrast to shear flow where reference is normally made only to steady shear flow behaviour, extensional flow behaviour is best described as being transient. In describing the transient behaviour of materials in extension at constant strain rate, they may exhibit either an unbounded stress growth behaviour in which the stress continually increases with increasing strain until the material fails, or the stress reaches a steady value with increasing strain thus yielding a tensile or equilibrium extensional viscosity. The latter occurs typically at large strains. An equilibrium extensional viscosity is thus dependent on strain rate but not on strain or time. Normally, the extensional viscosity will vary as a function of both strain and strain rate as well as temperature.

In describing and modelling plastics processing, the use of Hencky strain is preferred. The rate of Hencky strain of an element of fluid within a flow is independent of its original length and is determined only from the velocity field of that element. It is thus a more suitable characteristic of the flow. Strain and strain rate are taken by default herein to be Hencky values.

Stretching flow methods can be used to generate quantitatively accurate data on the extensional viscoelasticity of polymer melts. In carrying out extensional flow measurements, there are four types of measurement that are normally made: constant strain rate, constant stress, constant force and constant speed. This International Standard describes the first of these: constant strain rate. In this method, the strain rate is uniform throughout the specimen and is held constant with time.

The basic principle behind stretching flow measurements is to subject a specimen to a tensile stretching deformation. By measurement of the force and deformation of the specimen, the stresses and strains and hence strain rate can be determined.

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5 Apparatus

5.1 General description

The measuring apparatus shall consist of one of the following types, shown in Figures 1 to 4. These types define the various instrument configurations. The notation used in these figures is defined in 8.1.

Type A: Two rotating clamps. Each clamp shall consist of either a single rotating element or a pair of rotating elements — only the pair arrangement is shown. The force exerted on the specimen can be measured at the fixed or rotating end.

NOTE It is likely that the force will be easier to measure, and will be measured with greater accuracy, on a fixed clamp rather than on a moving clamp as there will be fewer complications due, for example, to vibration and the inertia of the clamp which may introduce noise and errors into the force signal.

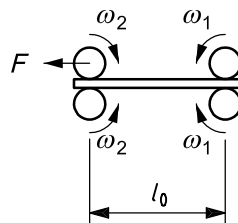


Figure 1 — Schematic diagram of type A test instrument

Type B: A single rotating clamp and a fixed clamp. The rotating clamp shall consist of either a single rotating element or a pair of rotating elements. The force exerted on the specimen is normally measured at the fixed end.

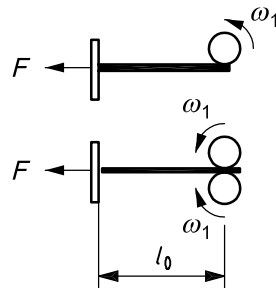


Figure 2 — Schematic diagram of type B test instrument

Type C: Two translating (non-rotating) clamps.

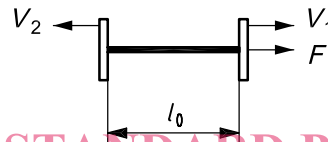


Figure 3 — Schematic diagram of type C test instrument

Type D: Single translating (non-rotating) clamp.

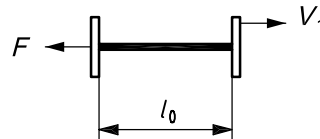


Figure 4 — Schematic diagram of type D test instrument

In each of these configurations, the specimen is mounted between the clamps and is stretched uniaxially. The requirements for the apparatus are that it shall permit the measurement or determination of the force acting on the specimen, and the strain and strain rate of the specimen subjected to a constant strain rate under isothermal conditions. The strain and strain rate of the specimen shall either be derived from the displacements and/or speeds of the clamp or clamps, or be measured directly from the dimensions and/or local velocities of the specimen.

5.2 Silicon bath/temperature-controlled chamber

Heating may be provided by placing the specimen in a silicone oil bath or in a temperature-controlled chamber with a forced gas flow through it.

NOTE 1 When heating using forced gas, a gas may be used in the chamber surrounding the test specimen to provide the required test environment, for example nitrogen to provide an inert atmosphere.

NOTE 2 The use of a silicone oil bath may permit more rapid heating of the specimen.

For low-viscosity materials, it is essential to support the specimen during heat-up and testing (to avoid it sagging under the influence of gravity).

NOTE 3 The use of a silicone oil bath results in the specimen being supported by the silicone oil due to its buoyancy, particularly if the densities of the silicone oil and specimen are matched at the test temperature. If a forced-gas oven is used, then support of the specimen can be obtained by the cushioning effect provided by the gas.

Silicone oil may be absorbed by some polymers. A check should preferably be made to see if the immersion time affects the measured properties of the polymer by varying the length of the immersion time (see Annex A and also Note 1 in 7.1). When quantitative results are required, then this check shall be made.

NOTE 4 Even if the silicone oil does not affect the shape of the tensile stress growth coefficient versus strain (or time) plot, it may affect the point of failure of the specimen. Thus assessment of the effect of the silicone oil on the measured properties should consider both of these aspects.

NOTE 5 Alternative methods for checking the effect of immersion in silicone oil on the specimen may also be used. Such methods include the measurement of the mass or dimensions of the specimen before and after immersing in silicone oil and identifying whether a change has occurred due to that immersion — see Annex A.

5.3 Temperature measurement and control

The test temperature should preferably be measured using a device that is mounted close to the specimen. Contact of the device with the specimen is not permitted. It is essential to mount temperature sensors in at least two positions to monitor temperature uniformity along the length of the specimen.

NOTE The uniformity of the temperature along the specimen length is critical to the measurement of the transient extensional flow properties of polymer melts. Localized hot spots will result in excessive strain in those regions. This may lead to premature failure, particularly for materials that do not exhibit a high degree of strain hardening.

The spatial temperature variation shall be within $\pm 0,75$ °C.

The temporal temperature variation shall be within $\pm 1,0$ °C of the set temperature.

The temperature-measuring device shall have a resolution of 0,1 °C and shall be calibrated using a device accurate to within $\pm 0,1$ °C.

5.4 Strain and strain rate measurement

The strain and strain rate of the specimen shall be determined either from measurement of the displacements and/or speeds of the clamp or clamps, or measured directly from the dimensions and/or local velocities of the specimen.

NOTE The diameter of the specimen may be measured during the test by use of optical or cutting methods to derive strains and strain rates and to assess the uniformity of deformation. The cutting method results in the test being terminated once the cuts have been made and thus prevents data to failure from being obtained. Local velocities may be measured using optical methods.

Corrections for slippage of the specimen at the clamp or clamps may be applied, obtained through independent measurement of the strain of the specimen during testing through measurement of its diameter or local velocities by other methods.

The apparatus shall have an accuracy of strain determination or measurement to within ± 3 % of the absolute value.

The apparatus shall have an accuracy of strain rate determination or measurement to within ± 3 % of the absolute value.

5.5 Force measurement

The force on the specimen shall be measured during the test by an appropriate means, for example a leaf spring arrangement (see Note 1).

The resolution of the force-measuring device should preferably be no greater than 0,1 % of the full-scale value.

The apparatus shall have an accuracy of force measurement to within ± 2 % of the full-scale value (see Note 2).

NOTE 1 Typical peak forces measured in testing of polyethylenes are estimated to be up to ≈ 1 N for specimens approximately 3 mm in diameter.

NOTE 2 It is desirable, in particular for accurate measurements at low forces, that the accuracy of the force measurement device be within ± 2 % of absolute, but this may be difficult to achieve in the lower part of the force transducer's range.

5.6 Calibration

The force, displacement, rate of displacement and temperature functions of the rheometer shall be calibrated periodically.

It is preferable that calibration be carried out at the test temperature as measurement of these functions, in particular that of force, may be temperature sensitive.

NOTE No traceable standard reference materials are known to exist for checking the calibration of such instruments. Where a reference material is used for checking the instrument, it is preferable that the transient extensional viscosity of the reference material, and the dimensions of the specimen produced using it, have values that are similar to those encountered or used during normal operation of the instrument.

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6 Sampling and specimen preparation

6.1 Sampling

The sampling method, including any special methods of specimen preparation and introduction into the rheometer, shall be as specified in the relevant materials standard or otherwise by agreement.

If samples or specimens are hygroscopic or contain volatile ingredients, then they shall be stored to prevent or minimize any effects on the measurements. Drying of samples may be required prior to preparing test specimens.

As the test specimens are typically small, being of the order of a few grams, it is essential that they be representative of the material being sampled. Repeat testing may be used to identify batch-to-batch or within-batch variation.

6.2 Specimen preparation

The specimen shall be either cylindrical or rectangular in cross-section.

Test specimens in the form of a cylinder may be produced by extrusion or by injection, transfer or compression moulding.

Test specimens in the form of a strip may be produced by extrusion or by injection or compression moulding or by cutting from sheet.

The length-to-diameter ratio of cylindrical specimens should preferably be at least 10.

NOTE A length-to-diameter ratio of at least 10 is required to minimize end-effects. However, a longer specimen will result in a reduction in the maximum strain rate that can be achieved. The magnitude of the end-errors can be assessed by using specimens of different length or diameter to produce different length-to-diameter aspect ratios. The effect on measured values can then be determined.

The specimen shall not contain any visible impurities, voids or air bubbles. The specimen shall not show any obvious discoloration prior to or after testing.

For cylindrical specimens, measure the diameter D of the specimen at at least three positions along its length. Repeat these measurements after rotating the specimen by 90° . Calculate an average value for the diameter from these measurements.

For rectangular specimens, measure the width and thickness at at least three positions along its length. Calculate average values for the width b and thickness h from these measurements.

Calculate the cross-sectional area of the specimen from the measurements.

The diameter, or width and thickness, of the specimen, as appropriate, shall be determined to and be uniform to within $\pm 2\%$ of their average value.

6.3 Specimen mounting

Specimens may be either gripped by the clamps or attached using adhesive to studs that are then clamped into the instrument.

Attachment by a suitable high-temperature epoxy adhesive has been found suitable. Treat the ends of the specimen by passing them through a butane flame and then dipping them into concentrated sulfuric acid for 30 s. Prevent any other part of the specimen, except that to be bonded, from being exposed to either the flame or the acid. Dip the ends of the specimen into the adhesive and then attach them to the studs. Place the specimen with its studs into an oven and cure the adhesive using a suitable time-temperature cycle (100 °C for 1 h has been found suitable). Allow the specimen to cool before handling.

7 Procedure

7.1 Specimen loading

Mount a specimen in place in the rheometer.

Measure the length of the specimen between the clamps to within 1 % of its absolute value.

After mounting the specimen in the instrument, immerse it in the silicone oil bath or place it in the temperature-controlled chamber (see 5.2). Where possible, bring the bath or chamber to the test temperature before inserting the specimen to reduce the time spent by the specimen reaching and equilibrating at the test temperature. Allow the specimen and apparatus to reach thermal equilibrium at the test temperature. This period of time is referred to as the equilibration time.

NOTE 1 The adequacy of the time allowed for the specimen to reach thermal equilibrium and the effects of the silicone oil, degradation, crosslinking and other time-dependent phenomena on the specimen can be checked by varying the time for which the specimen is in the oil bath or environmental chamber before testing. The effect on the measured values can then be assessed. For measurements in silicone oil of specimens approximately 3 mm in diameter, an equilibration time of approximately 5 min has been found to be sufficient for testing at a temperature of 150 °C.

A correction for thermal expansion of the specimen during heating may be necessary. A correction for effects arising from stress relaxation of the specimen, if unclamped during the temperature equilibration period, may also be required. Both of these effects may result in a change in the critical dimensions of the specimen (i.e. thickness and width, or diameter) that may need to be taken into account.