SLOVENSKI STANDARD
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Ugotavljanje sipkosti in uporabnosti viskoelastičnih lepil z uporabo oscilacijske reometrije

Determination of the flowability and application behaviour of viscoelastic adhesives using the oscillatory rheometry

Bestimmung des Fließ- und Applikationsverhaltens von viskoelastischen Klebstoffen mit Hilfe der Oszillationsrheometrie

Détermination de l'attitude du fluage et de l'application des adhésifs viscoélastiques avec la méthode de la rhéologie oscillométrique

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 17408:2019) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, the secretariat of which is held by UNE.

This document is currently submitted to the CEN Enquiry.

SAFETY WARNING — Persons using this document shall be familiar with normal laboratory practice. The standard cannot address all safety problems that may be associated with its application. It is the responsibility of the user to define measures for health and safety at work and ensure that these correspond with the European and national regulations.

ENVIRONMENTAL PROTECTION NOTE — The materials approved in this standard can have negative effects on the environment. As soon as technological progress leads to better alternatives to these materials, they shall be removed from the standard as far as possible. At the end of the test, the user shall ensure a suitable disposal of the waste corresponding to the regional conditions.

1 Scope

This document specifies a measuring method for the characterization of rheological properties of structural adhesives using oscillatory rheometry. The advantage of the method in comparison to rotational viscometry measurements lies in the separation of elastic and viscous material properties, thus allowing to define the viscoelastic properties. This enables more precise information concerning the flow behaviour of the materials, thereby resulting in a better understanding of their processing properties.

The method described is particularly suitable for filled and paste-like adhesives. These are frequently processed using automated pump and application systems in industrial applications and will be set precisely considering their rheological properties. As the rheological behaviour of uncured adhesives is mostly independent of their properties in the cured state, the standard can also serve for the examination of non-structural adhesives.

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.

EN 923, *Adhesives — Terms and definitions*

DIN 53019-1, *Viscometry — Measurement of viscosities and flow curves by means of rotational viscometers — Part 1: Principles and measuring geometry*

DIN 53019-2, *Viscosimetry — Determination of viscosity and flow curves with rotational viscosimeters — Part 2: Viscosimeter calibration and determination of the uncertainty of measurement*

DIN 1342-1, *Viscosity — Part 1: Rheological concepts*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in DIN 53019-1, DIN 53019-2, EN 923 and DIN 1342-1 and the following apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
shear deformation

γ

relation of deflection s to the distance between the plats geometry or gap width H of a sample located between two plates at linear deflection of the upper plate in accordance with the tangent of the angle of deflection φ (see Figure 1)

$$\gamma = s / H = \tan(\varphi) \quad (1)$$

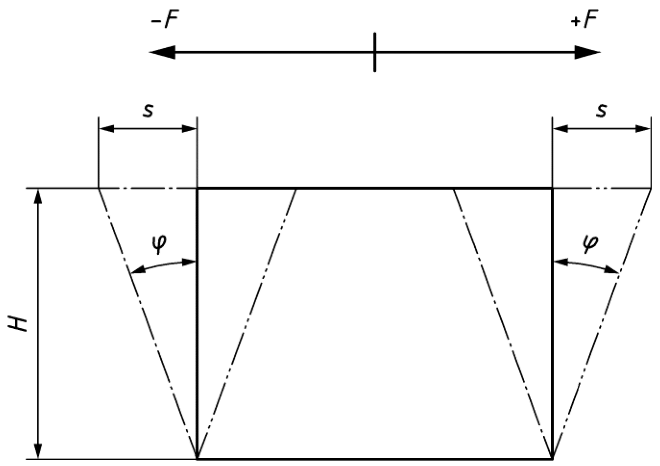

Figure 1 — Deflection *s* and angle of deflection *φ* of the test portion in the shear gap *H* [7]

Note 1 to entry: For a circular deflection in the plate/plate measuring system of a rheometer, this relation only applies for an infinitesimal surface element. The deflection here depends on the distance to the axis of rotation and is hence not uniform within the shear gap. The deformation value is, therefore, usually related to the plate edge (i.e. to $r_{\max}$), sometimes also to a mean distance to the axis of rotation (e.g. 2/3 $r_{\max}$). In this document, the plate edge is used as a reference value (as recommended in DIN 53019-1). The cone/plate configuration yields a constant deformation based on the gap width *H* raise equivalent to the deflection *s* increasing outwards in the entire shear gap.

3.2
deformation function

$\gamma(t)$

mathematical representation of the sinusoidal change in the deformation during oscillatory tests with controlled deformation

$$\gamma(t) = \gamma_A \sin(\omega t) \tag{2}$$

where

$\gamma(t)$ is the deformation at the time point t;

γ_A is the maximum deformation (deformation amplitude);

f is the frequency, in Hz;

ω is the angular frequency, in rad/s, with $\omega = 2\pi f$.

3.3
shear stress function

(t)

deformation as phase-shifted sinusoidal function of the shear stress related the response of the sample located in the gap (see Figure 2)

$$\tau\left(t\right) = \tau_A \sin\left(\omega\, t + \delta\right) \tag{3}$$

where

$\tau\ (t)$ is the shear stress at the time point t;

τ_A is the maximum shear stress (shear stress amplitude);

f is the frequency, in Hz;

ω is the angular frequency, in rad/s, with $\omega = 2\,\pi\,f$.

δ is the angle phase shift (loss angle).

Note 1 to entry: In the case of ideal-elastic behaviour (according to Hooke), the loss angle δ is 0°, i.e. deformation and shear stress are always in the same phase. Maximum shear stress is measured at maximum deformation. In the case of ideal-viscous behaviour (according to Newton), the loss angle δ = is 90°, i.e. the shear stress curve is ahead of the deformation curve by 90°. The maximum shear stress at deformation zero results here, i.e. at highest angular velocity of the test specimen.

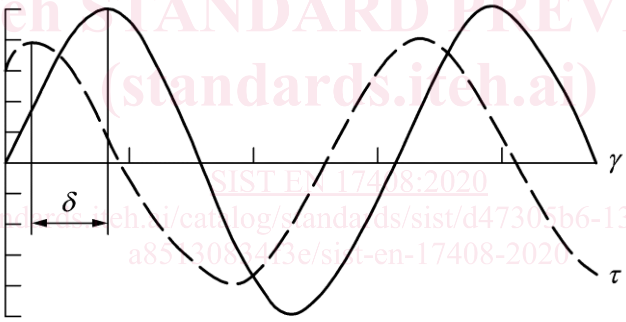

Figure 2 — Deformation and shear stress function during oscillation test [7]

3.4
storage modulus

G'

calculated from the energy stored during deformation, completely available for recovering after the end of the deformation process

$$G' = \frac{\tau_A}{\gamma_A} \cos\left(\delta\right) \tag{4}$$

where

τ_A is the maximum shear stress (shear stress amplitude);

γ_A is the maximum deformation (deformation amplitude);

δ is the angle phase shift (loss angle).

Note 1 to entry: The storage modulus represent the elastic portion of the applied energy and describes a typical solid property (solid like).

3.5
loss modulus

G”

calculated from the energy irreversibly consumed during the deformation, dissipated as heat

$$G'' = \frac{\tau_A}{\gamma_A} \sin(\delta) \quad (5)$$

where

τ_A is the maximum shear stress (shear stress amplitude);

γ_A is the maximum deformation (deformation amplitude);

δ is the angle phase shift (loss angle).

Note 1 to entry: The loss modulus represent the viscous (liquid like) portion of the applied energy dissipated as heat or work.

3.6
Complex shear modulus

*G**

vector sum of storage modulus *G′* and loss modulus *G”*

$$G^* = \tau(t) / \gamma(t) = G' + i \cdot G'' \quad (6)$$

$$\left|G^*\right| = \sqrt{G'^2 + G''^2} \quad (7)$$

where

$\tau(t)$ is the shear stress at the time point *t;*

$\gamma(t)$ is the deformation at the time point *t*;

G′ is the storage modulus;

G” is the loss modulus.

Note 1 to entry: When performing oscillatory tests on ideally elastic materials, i.e. completely inflexible, stiff and rigid solids, Hooke's Law applies, as indicated in Formula (6).

3.7
loss factor

tan (*δ*)

calculated as the quotient of loss modulus and storage modulus

$$tan(\delta) = G''/G' \quad (8)$$

where

G′ is the storage modulus;

G” is the loss modulus.

Note 1 to entry: The loss factor corresponds to the ratio between dissipated and reversibly stored deformation energy.

3.8
complex viscosity

η^*

ratio between the time – dependent values of the stress and deformation of shear rate

$$\eta^* = \tau(t) / \dot{\gamma}(t) \tag{9}$$

where

$\tau\ (t)$ is the shear stress at the time point t;

$\gamma\ (t)$ is the deformation or shear rate at the time point t.

Note 1 to entry: The complex viscosity is linked to the amount of the complex shear modulus via a simple relation:

$$|\ \eta^*\ | = \left|G^*\right| / \omega \text{ (with } \omega = 2\ \pi f\text{)} \tag{10}$$

Note 2 to entry: Similar to the complex shear modulus, the complex viscosity also comprises an elastic and a viscous component or better it is a sum of the real part η' and imaginary part η''.

3.9
linear-viscoelastic range

LVE range

range of low deformations, in which the amplitudes τ_A and γ_A are proportional to one another

Note 1 to entry: The quiescent structure of the substance is extensively retained in this range, and the deformations are reversible. The functions of G^*, G' and G'' (and likewise the viscosity variables linked to this) are constant and form a plateau value (see Figure 3). For many of the adhesives considered here, this is the case with deformation values less than or equal to 0,1 %. Besides the material situations, the LVE range also depends on the temperature and measuring frequency. Strictly speaking, the rheological relations indicated above only apply exactly in this range. However, the LVE range shall be occasionally departed from to describe the practical behaviour of adhesives.

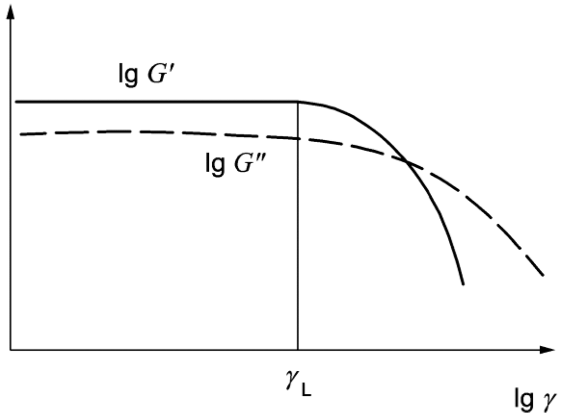

Figure 3 — G' and G'' as function of the deformation with the critical value γ_L of the linear-viscoelastic range [7]