

INTERNATIONAL STANDARD

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Glass-to-glass sealings — Determination of stresses

Soudure verre-verre — Détermination des contraintes



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 4790 was prepared by Technical Committee ISO/TC 48, *Laboratory glassware and related apparatus*, Sub-Committee SC 5, *Quality of glassware*.

Annex A of this International Standard is for information only.

Glass-to-glass sealings —Determination of stresses

1 Scope

This International Standard describes the test method for determining the stresses which may occur after the sealing of two glasses by means of stress birefringence.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 10345-1:1992, *Glass — Determination of stress-optical coefficient — Part 1: Tensile test.*

ISO 10345-2:1992, *Glass — Determination of stress-optical coefficient — Part 2: Bending test.*

FEPA-Standard 43-GB-1984,¹⁾ *Standard for coated abrasive grains of fused alumina and silicon carbide.*

3 Principle

If two glasses are sealed together, stresses can persist in them after cooling. The value of these stresses depends on the differences in the thermal, elastic and viscous properties of the glasses.

In general, glasses become birefringent when they are subjected to stresses. For the relationship between the stress and the optical path difference re-

sulting from the birefringence, the following equation applies:

$$\sigma = \frac{\Delta s}{aK} \quad \dots (1)$$

where

σ is the tensile or compressive stress;

Δs is the optical path difference;

K is the stress optical coefficient;

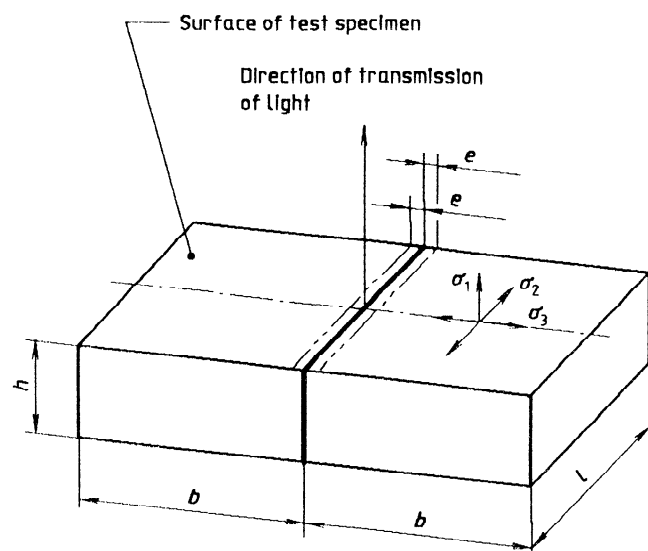
a is the light path in the test specimen (which is identical with the height h of the test specimen).

For positive stress-optical coefficients K , in the case of a tensile stress in the glass, the value for Δs shall have a positive sign (+), and in the case of a compressive stress in the glass a negative sign (−). For negative stress-optical coefficients K , the signs of Δs are the contrary.

It is assumed that a plane state of stress is set up predominantly in the test specimen and that the stress zone in the direction of transmission of light in the vicinity of the sealed area is approximately homogenous. The three principal stresses are orientated in such a way that

- the first principal stress σ_1 is perpendicular to the surface of the test specimen;
- the second principal stress σ_2 is parallel to the sealed area and to the surface of the test specimen;
- the third principal stress σ_3 is perpendicular to the sealed area (see figure 1).

1) FEPA: Federation of European Producers of Abrasive Products.

**Key**

- e = 0,5 mm (see 6.6)
 b Breadth of the glass pieces
 h Height of the test specimen and the glass pieces
 l Length of the test specimen and the glass pieces
 $\sigma_1, \sigma_2, \sigma_3$ Principal stresses in the test specimen

Figure 1 — Principal stresses in the test specimen

In equation (1), σ represents the difference between the principal stresses σ_2 and σ_3 . In the vicinity of the sealed area, the principal stress σ_3 is small in comparison with the principal stress σ_2 , so that in equation (1), σ may be treated as equivalent to principal stress σ_2 .

The optical path difference, which occurs when light passes between the two light waves oscillating in the direction of principal stresses σ_2 and σ_3 , is caused by the differing speeds of propagation in birefringent test specimens.

4 Apparatus

4.1 Furnace, for sealing and cooling the test specimen as described in 5.3.1 and 5.3.2.

4.2 Stress-testing equipment, for a survey method for testing the test specimen as described in 6.1 and 6.2.

4.3 Measuring device, capable of measuring the height h of the test specimen to 0,1 mm.

4.4 Polarization measuring equipment, with a compensator capable of measuring the optical path difference in the vicinity of zero setting to 5 nm. Depending on the table of functions of the compensator

used, either white light or a light source of spectral wave range between 540 nm and 590 nm is recommended (spectral lamps or incandescent lamps with an interference filter).

NOTE 1 The deviations between any optical path differences measured in the wave range between 540 nm and 590 nm are smaller than the uncertainty of the polarization measuring equipment.

5 Test specimen

5.1 Dimensions of glass pieces (see figure 1)

The two glass pieces to be sealed together shall be of equal size and shall comply with the following requirements:

- height h : 4 mm to 10 mm, preferably 5 mm;
- length l : 20 mm (approx.);
- breadth b : 10 mm (approx.);
- the corners and edges of the test pieces may be rounded.

5.2 Condition of glass pieces

The glass pieces shall be free of striae, gaseous and solid inclusions in the vicinity of the surfaces to be sealed.

The surfaces of the glass pieces to be sealed shall be flat, with a roughness less than that achieved by polishing with an abrasive of particle size P 240 according to FEPA-Standard 43-GB.

5.3 Preparation of the test specimen

5.3.1 Seal two glass pieces completely together, on the surfaces prepared for sealing, to form one test specimen (see figure 1). During this procedure, any deformation of the flat sealed surface shall be avoided.

5.3.2 Since the result of the measurement can be affected by the cooling rate applied to the test specimen in the visco-elastic range of the glasses, the test specimen shall be cooled at a rate of $(2 \pm 0,2) ^\circ\text{C}$ per min, within the following temperature range:

- the upper limit of temperature range is determined by the temperature at which the glass of the test specimen which has the higher viscosity has reached a dynamic viscosity of 10^{13} dPa s;
- the lower limit lies $150 ^\circ\text{C}$ below the temperature at which the glass with the lower viscosity has a dynamic viscosity of 10^{13} dPa s.

Outside this temperature range the only requirement is that temporary stresses occurring during cooling shall not cause splits in the test specimen.

5.3.3 After sealing, the surfaces of the test specimen through which the light passes (see figure 1) shall be treated so that they are parallel to each other and perpendicular to the sealed area.

6 Procedure

6.1 In order to verify that the sealing of the test specimen is without any defects (see 5.3.1), the symmetry of the stress picture should be checked in each half of the test specimen by means of stress-testing equipment (see figure 2).

6.2 In addition, both halves of the test specimen shall be tested for splits. Splits in one or both halves of the test specimens disturb the stress symmetry and this may affect the results of the measurements taken as described in 6.6. For such test specimens, only the sign of stress can be determined, as described in 6.7.

6.3 Take all measurements at room temperature between 15 °C and 35 °C. Any test temperatures differing from this range shall be stated in the test report.

6.4 Measure the thickness of the test specimen in the measuring area (see 6.6).

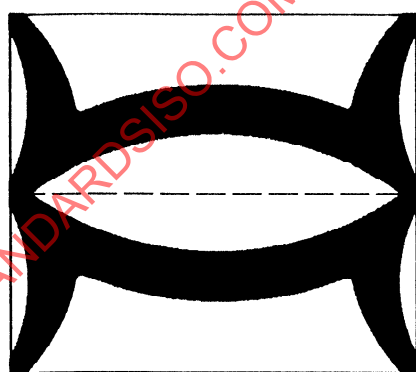
6.5 When the surfaces of the test specimens through which the light passes are not sufficiently smooth for the stress picture to be clear, it is advisable to place the test specimen in an immersion fluid, for example water in the case of fine polished surfaces. The bottom of the immersion vessel itself shall not have any interfering birefringence and optical path difference.

6.6 When the light is passed through a split-free test specimen in a perpendicular direction, measure the principal stresses lying parallel to both sides of the sealed surface as the optical path differences, one after another at a distance of $a = 0,5$ mm from the sealed surface and at the centre of the test specimen (see also figure 1).

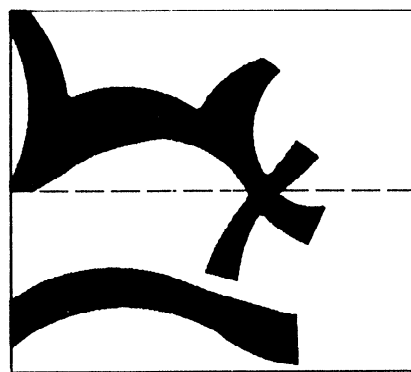
In the case of a test specimen with one non-transparent glass piece, the optical path difference can only be measured in the transparent half of the test specimen.

In the case of a test specimen of which one-half consists of an agreed reference glass, possibly of the same type of glass, it is sufficient to measure the optical path difference of the birefringence in the reference glass half.

6.7 When the type of stress is not clearly revealed by measurement of the optical path difference, and in the case of a test specimen having splits, determine by means of the stress testing equipment and by comparison with a tension or compression test piece, whether the optical path difference is the result of tensile or compressive stress.



a) Correctly sealed glass pieces
(test specimen A)



b) Partially sealed glass pieces
(test specimen B)

NOTE — The areas which appear dark have an optical path difference of zero (stress free) or a whole multiple of the medium wave-length used. Dark parts also occur at the points at which the direction of principal stress in the test specimen coincides with the axis of polarization.

Figure 2 — Isoclinic pictures between two crossed polarizers (light-field with linear polarization, the sealed area at 45° to the axis of polarization).

7 Expression of results

For each half of the test specimen for which the optical path difference has been measured according to 6.6, calculate the stress birefringence Δn from the numerical equation (2) and the principal stress σ_2 from the numerical equation (3):

$$\Delta n = \frac{\Delta s}{h} \quad \dots (2)$$

$$\sigma_2 = 0,1 \frac{\Delta s}{hK} \quad \dots (3)$$

where

- Δn is the stress birefringence, in nanometres per centimetre;
- Δs is the optical path difference, in nanometres (see clause 3 for information concerning the sign);
- K is the stress-optical coefficient, expressed in $10^{-6} \text{ mm}^2/\text{N}$, according to ISO 10345-1 or ISO 10345-2;
- h is the height, in centimetres, of the test specimen;
- σ_2 is the principal stress, in newtons per square millimetre.

8 Test report

The test report shall include the following information:

- a) reference to this International Standard;
- b) types and designations of the glasses sealed;
- c) cooling rate, if other than $(2 \pm 0,2) \text{ }^\circ\text{C per min}$;
- d) testing temperature, if outside the range $15 \text{ }^\circ\text{C}$ to $35 \text{ }^\circ\text{C}$;
- e) for each half of a split-free test specimen:
 - the stress birefringence Δn , in nanometres per centimetre, to the nearest 5 nm/cm ;
 - principal stress σ_2 , in newtons per square millimetre, to the nearest $0,1 \text{ N/mm}^2$, with the corresponding sign according to clause 3;
 - the stress-optical coefficient K , expressed in $10^{-6} \text{ mm}^2/\text{N}$, used for the calculation of the principal stress σ_2 ;
- f) for test specimens with splits, the type of stress (tensile or compressive).