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Adhesives — Determination of temperature dependence of shear strength

*Adhésifs — Détermination de la résistance au cisaillement en fonction
de la température*

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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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Adhesives — Determination of temperature dependence of shear strength

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This International Standard specifies methods for determining the temperature dependence of the shear strength of the adhesive or adhesive bond in adhesively bonded products.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 4587, *Adhesives — Determination of tensile lap-shear strength of rigid-to-rigid bonded assemblies*

ISO 6238, *Adhesives — Wood-to-wood adhesive bonds — Determination of shear strength by compressive loading*

ISO 10365, *Adhesives — Designation of main failure patterns*

ISO 15605, *Adhesives — Sampling*

ISO 17212, *Structural adhesives — Guidelines for the surface preparation of metals and plastics prior to adhesive bonding*

3 Terms and definitions

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

3.1

temperature dependence of shear strength in adhesive bonds

property capable of being measured by determining the shear strength of test specimens at various temperatures and recording the changes

4 Methods of test for shear strength

4.1 Test method for metals

For adherends made of metal, the shear strength shall be determined in accordance with ISO 4587.

4.2 Test method for wood

For adherends made of wood, the shear strength shall be determined in accordance with ISO 6238.

5 Test atmosphere

The specimens shall be conditioned and tested in one of the standard atmospheres specified in ISO 291.

6 Sampling and handling of adhesives

The method used for sampling and for handling the adhesives shall be in accordance with ISO 15605.

7 Kinds of adhesive and surface preparation of adherends

See ISO 17212.

8 Preparation of test specimens

8.1 Metal adherends

The test joints may be prepared either individually or from adhesively bonded panels, in each case following the procedure specified in ISO 4587. Surface treatment of the adherends and application and curing of the adhesive shall be in accordance with the adhesive manufacturer's instructions.

Whether prepared individually or cut from adhesively bonded panels, the shape and dimensions of the specimens shall be as specified in ISO 4587.

For each test temperature (see Clause 9), the number of specimens tested shall be at least five.

8.2 Wood adherends

The test joints may be prepared either individually or from adhesively bonded blocks, in each case following the procedure specified in ISO 6238. Surface treatment of the adherends and application and curing of the adhesive shall be in accordance with the adhesive manufacturer's instructions.

Whether prepared individually or cut from adhesively bonded blocks, the shape and dimensions of the specimens shall be as specified in ISO 6238.

For each test temperature (see Clause 9), the number of specimens tested shall be at least 12 and, when they are cut from adhesively bonded blocks, they shall come from at least three blocks.

9 Procedure

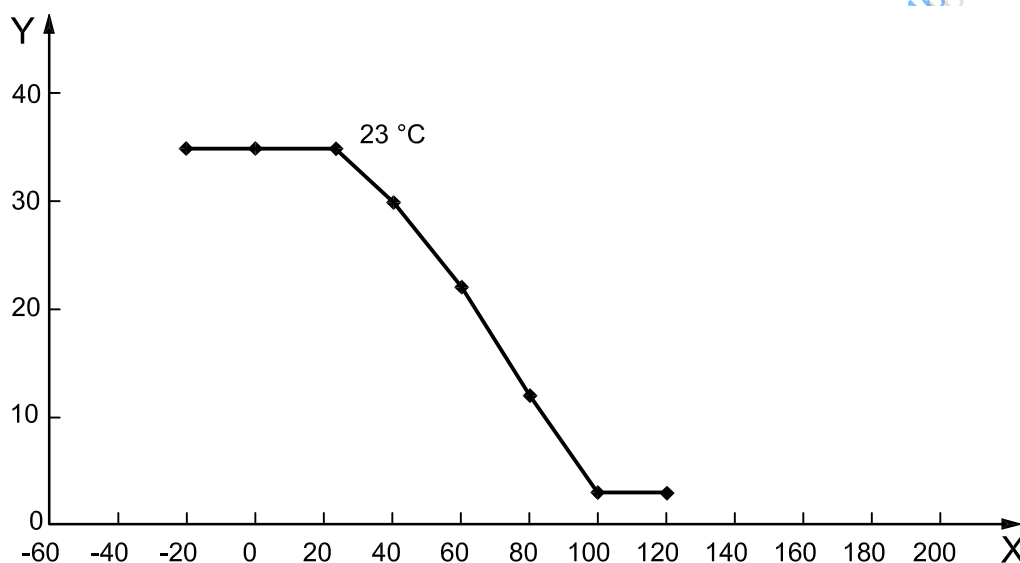
Immediately after being conditioned for 168 h (or 48 h in the case of heat-cured adhesives) in one of the atmospheres specified in ISO 291, specimens shall be tested in accordance with Clause 4 in the same atmosphere as used for conditioning.

In addition, specimens shall be tested at temperatures selected from the following: $(-40 \pm 3)^\circ\text{C}$, $(-20 \pm 3)^\circ\text{C}$, $(0 \pm 2)^\circ\text{C}$, $(40 \pm 2)^\circ\text{C}$, $(60 \pm 2)^\circ\text{C}$, $(80 \pm 2)^\circ\text{C}$, $(100 \pm 2)^\circ\text{C}$, $(120 \pm 2)^\circ\text{C}$, $(140 \pm 2)^\circ\text{C}$, $(160 \pm 2)^\circ\text{C}$ and $(180 \pm 2)^\circ\text{C}$, the additional conditioning time at each temperature being 10 min for metals and 24 h for wood. It is not necessary to control the humidity when testing at these temperatures.

10 Expression of results

10.1 Plot of shear strength versus temperature

Plot adhesive shear strength versus temperature using the results from Clause 9. Figure 1 shows an example of a typical plot.



Key

- X temperature ($^\circ\text{C}$)
Y shear strength (MPa)

Figure 1 — Typical plot of shear strength versus temperature

10.2 Failure patterns

Classify failure patterns in accordance with ISO 10365.

11 Test report

The test report shall include the following particulars:

- a reference to this International Standard;
- all details necessary for complete identification of the adhesive tested, including type, source, manufacturer's code number, batch or lot number, physical form, etc.;
- the shape and dimensions of the adherends, as well as the material of which they are made, their surface preparation and any other necessary information on them;
- the method used to apply the adhesive to the adherends, the drying and pre-cure conditions (if relevant), the curing time or setting time, and the bonding procedure used (including the bonding temperature and pressure);

- e) the average thickness of the bond after bonding and the method used to determine the thickness;
- f) details of test specimen preparation, the shape and dimensions of the test specimens, their number and construction, the atmosphere in which they were conditioned before testing and the atmosphere in which they were tested;
- g) the test speed (or the rate of loading in the case of constant rate of load application);
- h) the shear strength of each test specimen tested at each temperature, the mean shear strength and the standard deviation from the mean, and the plot of adhesive strength versus temperature;
- i) the failure patterns, classified in accordance with ISO 10365;
- j) details of any deviations from the specified procedure and of any incidents which may have affected the results;
- k) the date of testing.

Annex A (informative)

Practical information on the test method

A.1 General

This International Standard is relevant to adhesives that are used for the manufacture of products which are utilized in the interior of buildings. For these applications, a knowledge of the dependence of the adhesive strength on temperature is a useful indicator of the durability of the adhesive, and a measure of the temperature dependence of shear strength is an important aid in design and material selection.

This annex gives practical information relating to this standard, including background information on the method used as well as the reasons why this method was preferred to other methods.

There are three commonly used ways of determining the dependence of adhesive strength on temperature:

- by determination of the temperature dependence of the adhesive bond (i.e. the measurement of the adhesive strength at different temperatures)^[1];
- by determination of the thermal-degradation properties of the bond;
- by determination of the temperature dependence of the creep properties of the bond^[2].

This International Standard uses approach a).

A.2 Background information

A.2.1 Test methods not considered appropriate for this International Standard

A.2.1.1 Determination of adhesive strength at the glass-transition temperature

Since the mechanical properties of an adhesive change most rapidly at temperatures in the region of the adhesive's glass-transition temperature T_g [3], [4], [5], consideration has been given to characterizing temperature dependence by presenting strength results at T_g . Following experimental studies in which differential scanning calorimetry (DSC) and thermomechanical analysis (TMA) were employed to measure T_g , this approach was considered unsatisfactory for the following reasons:

- Some adhesives, such as epoxy resins and urethane resins, show extreme changes in adhesive strength around T_g , whereas others do not. This difference in behaviour is due to the fact that each resin has different softening properties.
- In the case of emulsion resins, it is very difficult to measure T_g because of the effects of the emulsifying agent and the solvent.
- The reproducibility of determinations at T_g is problematic because the value of T_g depends on the method used to measure it, such as DSC, TMA or differential thermal analysis (DTA), as well as on the operator carrying out the measurement and the measurement conditions (rate of heating).

A.2.1.2 Classification of adhesives by temperature dependence

Next, a method for classifying adhesives by measuring the reduction in strength at an elevated temperature was explored.

The adhesive strength of adhesively bonded products, or parts of such products, was measured at 23 °C and at a higher temperature between 23 °C and 100 °C. The adhesives were then classified into four types

according to the value of the adhesive strength at the elevated temperature expressed as a percentage of the adhesive strength at 23 °C.

At this stage, it was recognized that the classification of adhesives in this way was problematic, as it does not make sense to classify adhesives whose adhesive strength at 23 °C is totally different from each other as the same type.

Another approach was needed, for example:

- ranking adhesives at a single temperature, e.g. 80 °C, which is a suitable temperature for assessing temperature dependence;
- determining the maximum temperature up to which the adhesive retains the adhesive strength it has at 23 °C.

Approaches a) and b) were rejected, however, because they were of limited applicability.

In conclusion, instead of presenting data in the way shown in Table A.1, plotting a graph of adhesive shear strength versus temperature has been adopted in this International Standard (see Figure 1). Adhesive manufacturers and users can easily understand adhesive properties with this kind of plot.

Table A.1 — Classification of adhesives by their temperature dependence

Type	Adhesive strength at an elevated temperature expressed as a percentage of the adhesive strength at 23 °C
	%
1	100 to 80
2	79 to 60
3	59 to 40
4	39 to 0

A.2.2 Method selected for this International Standard

A.2.2.1 Test specimens

As a result of investigation of several types of adherend — such as metal, glass, tile, concrete, mortar, plastics, reinforced plastics (FRP, etc.), wood and wooden material — thin metal plates and wood were selected for this method. These materials give good results in evaluating the durability of adhesives and, furthermore, are readily available. Test specimens cut out of adhesively bonded products were also tested.

A.2.2.2 Test temperature range

By means of questionnaires circulated to experts in the fields of plywood, wooden components, paper, cars, aircraft, shoes, electrical appliances, etc., it was established that the temperatures most commonly used for testing adhesively bonded products lie in the range from –40 °C to +120 °C.

However, since the temperature used can range from –40 °C to +180 °C, the range used in this International Standard has been fixed at –40 °C to +180 °C, with measurements at 20 °C intervals, as specified in ASTM D 1151 [6].

Bibliography

- [1] JIS K 6831, *Adhesives — Determination of temperature dependence of adhesive bonds*
- [2] JIS K 6859, *Testing methods for creep rupture of adhesive bonds*
- [3] ISO 3146, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods*
- [4] JIS K 7121, *Testing methods for transition temperatures of plastics*
- [5] JIS K 7122, *Testing methods for heat of transitions of plastics*
- [6] ASTM D 1151, *Standard Practice for Effect of Moisture and Temperature on Adhesive Bonds*

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