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Adhesives — Test methods for fatigue properties of structural adhesives in tensile shear

Adhésifs — Méthodes d'essai de tenue à la fatigue d'adhésifs structuraux en traction-cisaillement

Reference number
ISO 9664:1993(E)

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 9664 was prepared by Technical Committee ISO/TC 61, *Plastics*, Sub-Committee SC 11, *Products*.

Annexes A and B form an integral part of this International Standard.

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Adhesives — Test methods for fatigue properties of structural adhesives in tensile shear

1 Scope

This International Standard specifies a method for estimating the fatigue strength of adhesives in shear by tension loading, using standardized specimens under specified conditions, with the aim of characterizing structural adhesives on a given metallic substrate.

The fatigue properties are a function of the specimen geometry. The results do not correspond to intrinsic properties of the adhesive and cannot be used for design purposes.

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 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 4587:1979, *Adhesives — Determination of tensile lap-shear strength of high strength adhesive bonds.*

ISO 4588:1989, *Adhesives — Preparation of metal surfaces for adhesive bonding.*

3 Definitions and symbols

For the purposes of this International Standard, the following definitions and symbols apply.

3.1 shear stress (τ)**:** Stress determined by dividing the force by the bonded surface area.

It is expressed in megapascals (MPa).

3.2 static shear strength (τ_R)**:** Average static shear stress at rupture as determined by ISO 4587.

It is expressed in megapascals (MPa).

3.3 stress cycle: Smallest part of the stress/time function which is repeated at regular intervals.

It is of sinusoidal form (see figure 1) with undulating shear.

Cyclic stress may be considered to be the superposition of an alternating stress on a static stress which is the mean stress.

3.3.1 maximum stress (τ_{max})**:** Greatest algebraic value reached at regular intervals by the stress.

It is expressed in megapascals (MPa).

3.3.2 minimum stress (τ_{min})**:** Smallest algebraic value reached at regular intervals by the stress.

This stress shall always be positive and is expressed in megapascals (MPa).

3.3.3 mean stress (τ_m)**:** Albegraic mean of the maximum and minimum stresses.

$$\tau_m = \frac{\tau_{max} + \tau_{min}}{2}$$

It is expressed in megapascals (MPa).

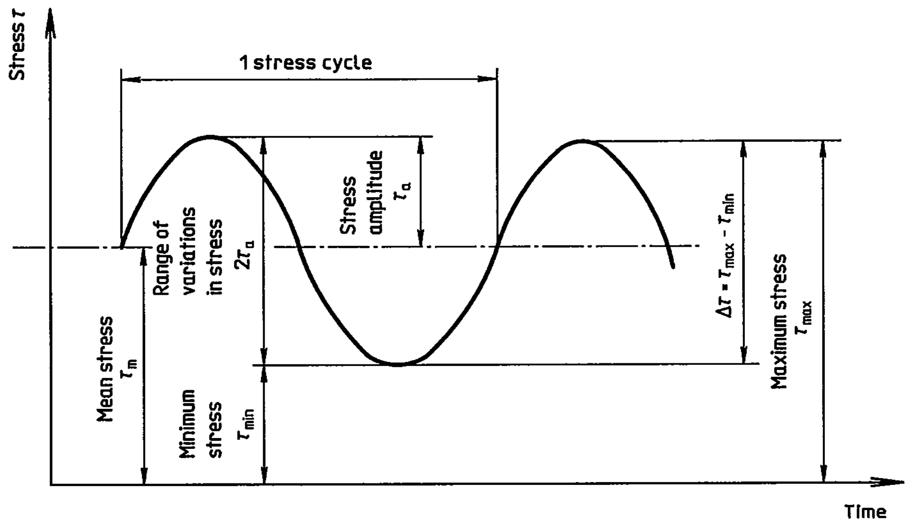


Figure 1 — Fatigue stress cycle

3.3.4 stress amplitude (τ_a)**:** Alternating stress equal to half the algebraic difference between the maximum and minimum stresses.

$$\tau_a = \frac{\tau_{max} - \tau_{min}}{2}$$

It is expressed in megapascals (MPa).

3.3.5 stress ratio (R_τ)**:** Algebraic ratio of the minimum stress to the maximum stress in one cycle.

$$R_\tau = \frac{\tau_{min}}{\tau_{max}}$$

3.4 fatigue limit (τ_D)**:** Limiting value which the stress amplitude τ_a approaches when the number of cycles becomes very large, for a given mean stress τ_m or stress ratio R_τ.

For some materials, stress amplitude versus the number of cycles does not reach a limiting value but decreases constantly on increasing the number of cycles. In this case it is useful to determine a limit of endurance.

3.5 limit of endurance [$\tau_D(N_F)$]**:** Shear stress determined at a specific number of fault test cycles N_F.

It is expressed in megapascals (MPa).

Depending on whether the tests are carried out at constant τ_m or at constant R_τ, the results should be presented in the form:

$\tau_D(N_F, \tau_m)$ in megapascals (MPa)

or

$\tau_D(N_F, R_\tau)$ in megapascals (MPa)

3.6 service life (N)**:** Number of stress cycles applied to a specimen until it has reached the chosen end of the test. Where it has not failed, the service life is not defined but is termed greater than the test duration.

3.7 cycle ratio (n/N)**:** Ratio of the number of applied cycles (n) to the service life (N). This ratio is used in tests with load bearings, together with an SN curve (Woehler's curve).

3.8 SN curve: Curve, allowing the resistance of the material to be seen, which indicates the relationship observed experimentally between service life N, shown conventionally in abscissae (logarithmic scale) and stress τ_a or τ_{max} shown in ordinates in linear scale [typical curve in figure 2 a)] or in logarithmic scale [typical curve in figure 2 b)]. This curve is established by keeping either τ_m or R_τ constant. The SN curve is defined by the relationship between amplitude of stress and service life. On this curve [figure 2 a)] we can distinguish:

— the endurance zone where, for a given stress, failures as well as non-failures for a number of fault test cycles N_F can be identified;

— the fatigue zone where, for a given stress, all the specimens fail at the end of a number of cycles less than the number of conventional fault test cycles N_F mentioned above.

4 Principle

The specimen is cyclically stressed in a way that may be regarded as the superposition of an alternating stress on a static stress which is the mean stress.

The number of cycles at failure of the specimen is determined for a given τ_m and τ_a. These values are used to establish SN curves which then permit the estimation of the confidence zone concerning the fatigue resistance of a joint.

5 Apparatus

5.1 Template, for the accurate positioning of substrates during bonding.

5.2 Fatigue test machine, to enable sinusoidal fatigue stress cycles to be obtained such that the maximum stress is between 10 % and 80 % of the scale range. The test frequency and the type of equipment may affect the test result. Unless indicated otherwise, the frequency shall be 30 Hz. The maximum frequency shall be 60 Hz, since excessive heating of the bond can occur at frequencies higher than 60 Hz. The machine shall be provided with a self-centring device for attachment of the specimen. The device shall be designed in such a way that its various components move in perfect alignment with the specimen as soon as the specimen is subjected to stress. In this way the major axis of the specimen coincides with the direction of application of the force and the axis of symmetry of the device.

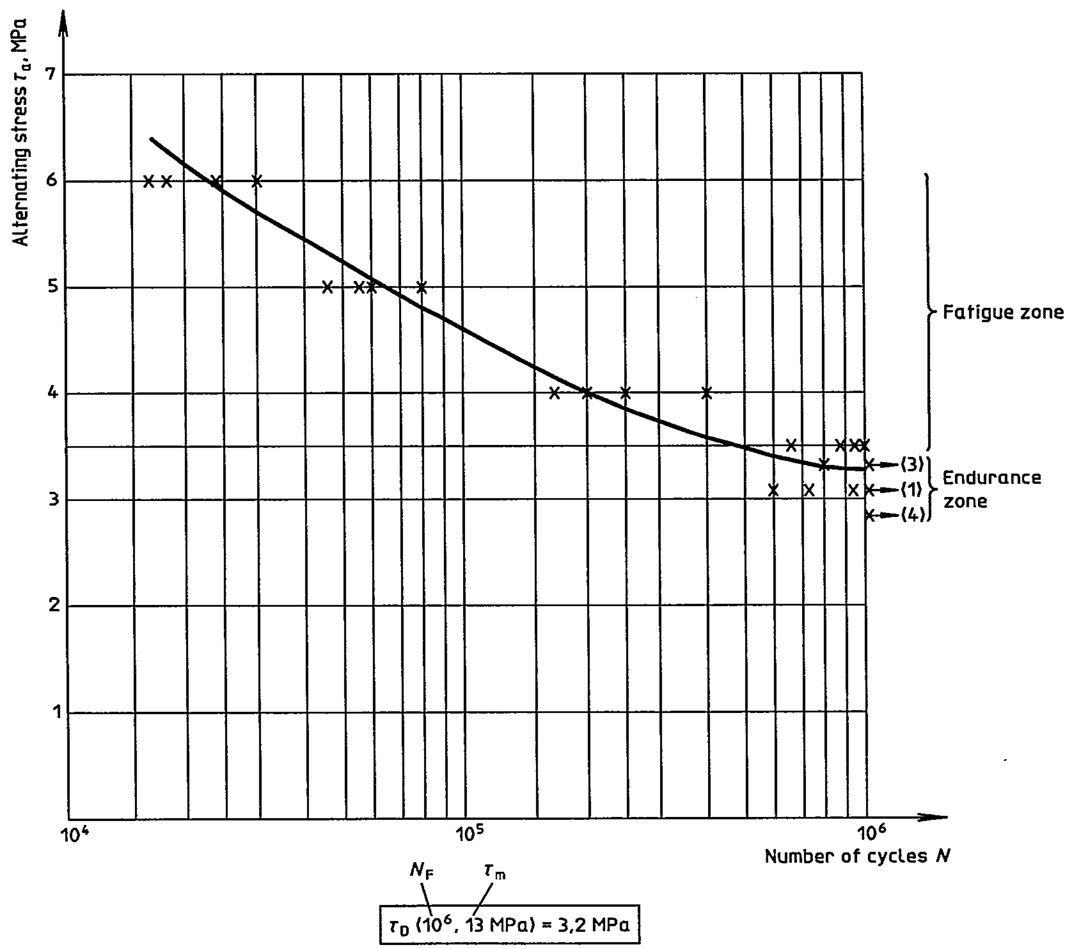
Attachments passing through the substrate may be used, in which case the specimen shall be reinforced as indicated in figure 3 b), using additional supports.

6 Specimens

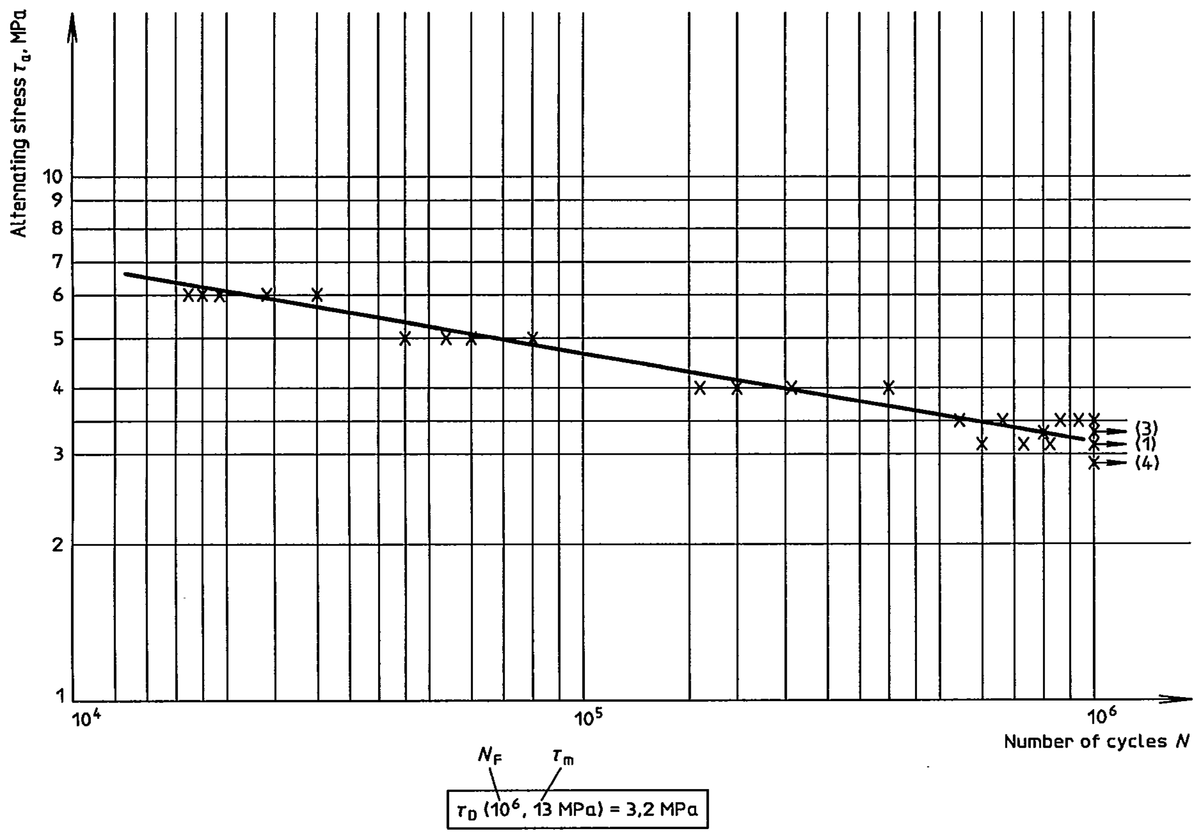
6.1 Substrate materials

The specimens shall conform in shape, dimensions and alignment to those indicated in figures 3 a) or 3 b) for steel and 3 c) for aluminium.

NOTE 1 Recommended substrates are aluminium 2024 A 5T3 and steel XC 18 or E 24, grade 1 or 2. Other grades may be used, depending on the end use of the adhesive.



2 a) Standard steel specimens, test at 30 Hz at room temperature, semi-logarithmic coordinates



2 b) Standard steel specimens, test at 30 Hz at room temperature, log-log coordinates

Figure 2 — Typical SN curve of a one-component epoxy adhesive

6.2 Preparation

Prepare the bonded specimens individually or from panels which may or may not be grooved (see figure 4).

In selecting the method of preparation, account shall also be taken of the possible mechanical damage to the bonded specimens. Special attention shall be paid during the preparation of individual specimens to achieving a suitable alignment and good homogeneity of the joint, particularly with regard to its thickness.

Surface preparation shall be carried out in accordance with ISO 4588, unless otherwise specified. The adhesive shall be used in accordance with manufacturer's instructions. In all cases the use of a template is necessary to ensure correct overlap, accurate alignment of the substrates, and regular thickness of the bonded joint for each specimen and for collections of specimens.

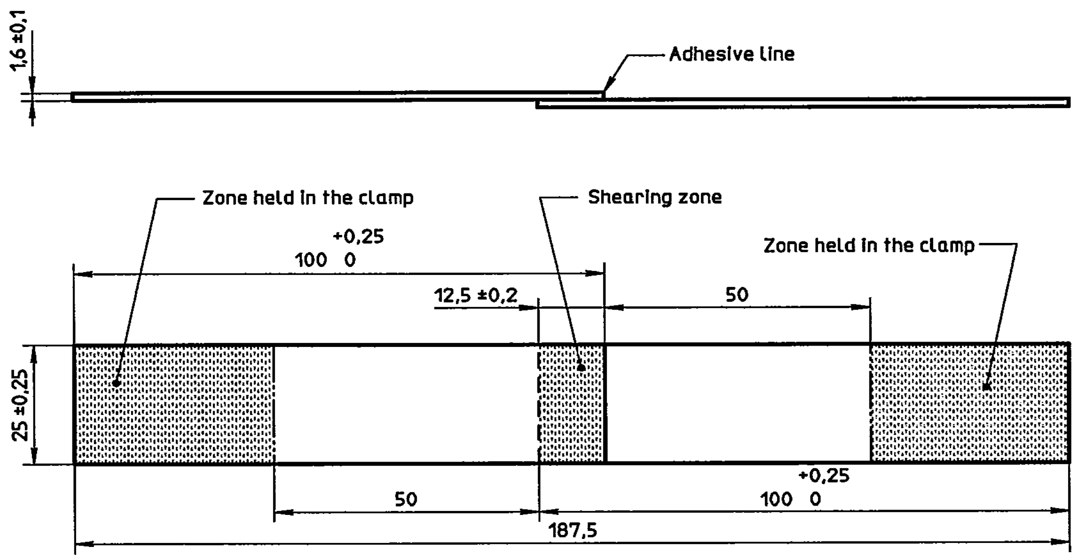
6.3 Number of specimens

The number of specimens depends on the precision required and the results sought. The minimum number of specimens is defined as follows:

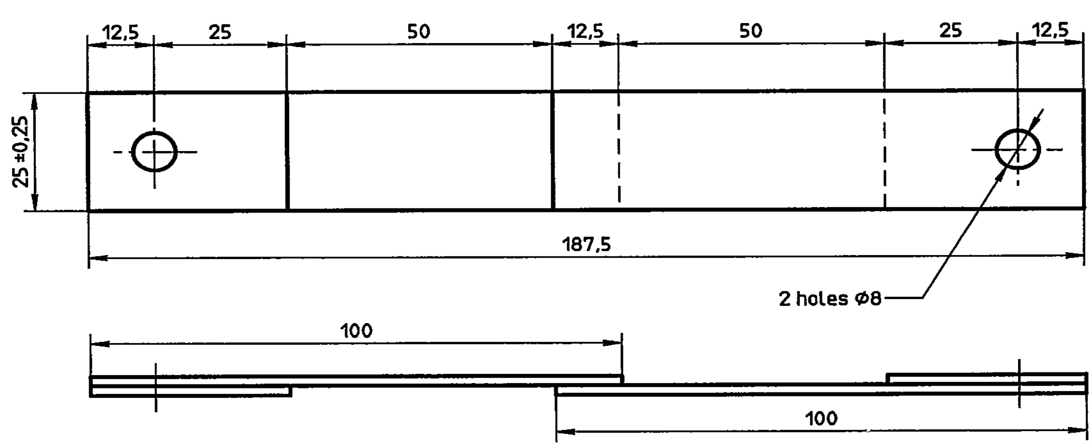
— at least four specimens tested at three different values of τ_a such that failure occurs between 10^4 and 10^6 cycles. This allows at least 12 specimens for the statistical determination of the limit of endurance at 10^6 cycles;

— at least six specimens for the determination of the static shear strength τ_R.

Dimensions in millimetres

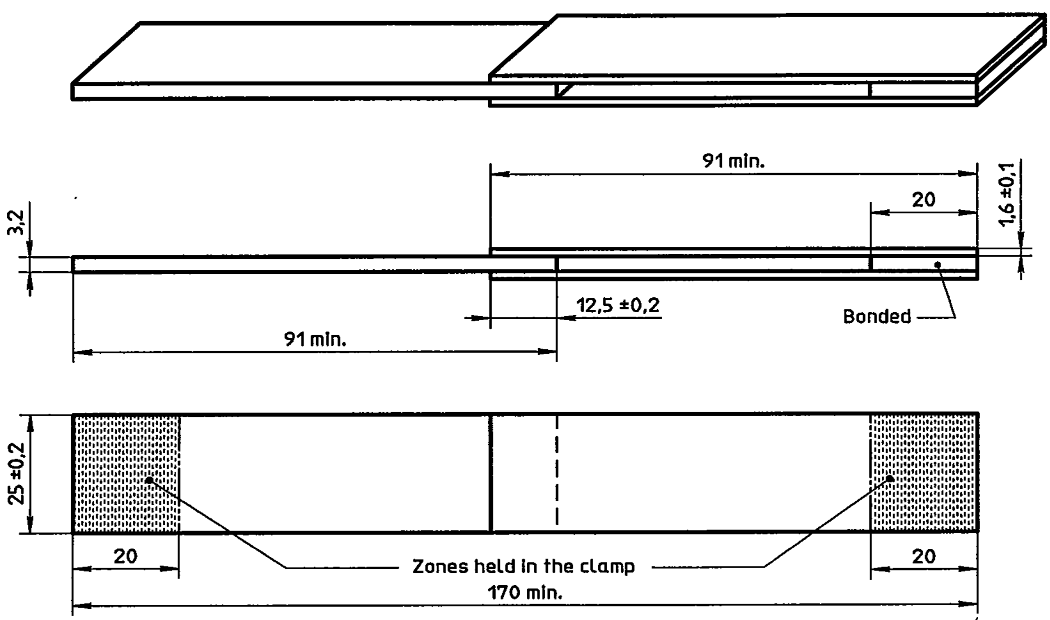


3 a) Steel substrates held in a clamp



3 b) Steel substrates fixed across supports

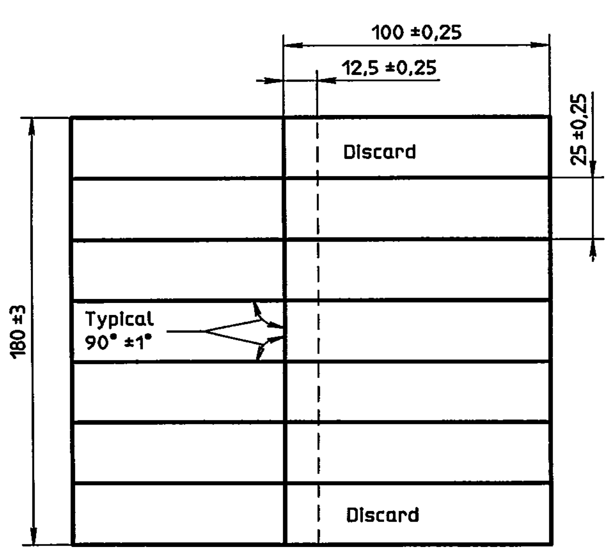
Dimensions in millimetres



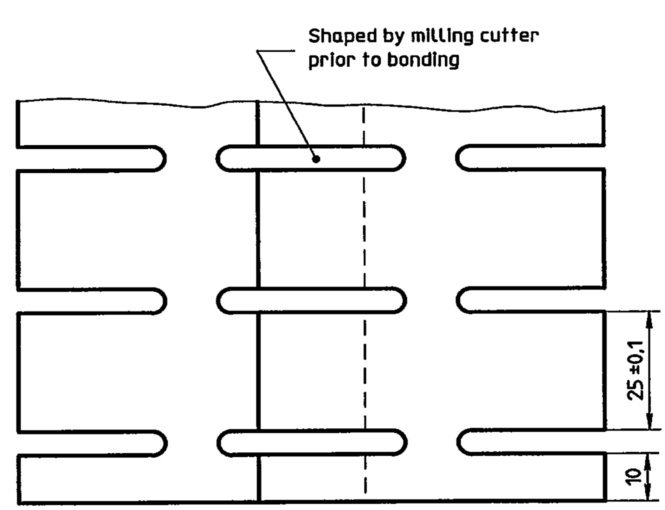
3 c) Aluminium substrates with double overlap

Figure 3 — Shape and dimensions of the specimen

Dimensions in millimetres



4 a) Typical test panel for steel



4 b) Typical test panel with double overlap for aluminium

Figure 4 — Test panels

6.4 Test conditions

The specimens shall be stored and subjected to test under the standard atmosphere of 23 °C ± 2 °C and (50 ± 5) % relative humidity as specified in ISO 291.

7 Procedure

7.1 General conditions

Adhesives may be subject to creep, even at ambient temperature, under the effect of a non-zero mean stress τ_m. Ensure before the fatigue test that the mean stresses used during the test period do not cause a failure due to creep to be attributed to a failure due to fatigue.

Fix the specimens symmetrically in the clamps at the distance indicated in figure 3, depending on the type of specimen tested.

Bring the specimen up to its mean stress τ_m and then up to the test frequency so that the amplitude τ_a is reached.

7.2 Construction of the SN curve, at a given mean stress value τ_m

Specimens shall be tested for fatigue properties after assessing the static shear strength τ_R on a lot of at least six specimens of the same configuration.

7.2.1 Select the τ_m value in the range 0,25 τ_R to 0,5 τ_R, where τ_R is the static shear strength of the adhesive, and indicate the chosen value in the test report.

NOTE 2 The τ_m value generally used is 0,35 τ_R.

7.2.2 Test at least four specimens for each of the amplitudes τ_a chosen such that the failures occur between 10^4 and 10^6 cycles. A minimum of three different amplitudes is necessary in this range. Determine the limit of endurance at 10^6 fault test cycles N_F.

In that part of the curve where the service life becomes very long, the scatter, at a given level of stress, results in failures. In such a case the results shall be indicated on the curves using a particular symbol, e.g.:

X for failed specimens, and

X → for specimens which have not failed [the number of specimens which have not failed is shown in brackets (see e.g. figure 2)].

In this region, the correct determination of a point on the SN curve requires the use of a statistical method.

Where the specimens are tested individually, the staircase method is used to determine the endurance limit $\tau_D(N_F, \tau_m)$ (see annex A).

If the fatigue machine used is equipped with a jig allowing the simultaneous testing of a group of specimens, the results are processed in accordance with the data reclassification method (see annex B).

7.2.3 Plot the SN curve, passing through the centre-line points and the endurance limit $\tau_D(N_F, \tau_m)$ with coordinates either (τ_a, lg N) or (lg τ_a, lg N) which permits a straight line to be obtained.

NOTE 3 A similar method may be used to obtain the SN curve at a constant R_τ value (generally 0,1) instead of a constant τ_m value.

8 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the time of revision.

9 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) all the information necessary for identification of the adhesive tested;

c) identification of the substrates;

d) method of surface preparation;

e) description of the methods of bonding and curing of the adhesive;

f) test conditions;

g) mean thickness per specimen of the adhesive layer, after the joint has been produced, including the accuracy of measurement;

h) number of specimens;

i) frequency used;

j) static shear strength τ_R of the adhesive;

k) results of τ_m, τ_D and τ_a, in megapascals, N (number of cycles) and the curves obtained in accordance with the procedure described in clause 7.

Annex A
(normative)

Staircase method (or Dixon and Mood's method)

A.1 Principle

A maximum test duration (number of cycles) and a staggering of stress amplitudes is defined, spaced out in accordance with arithmetic progression of ratio d (spacing close to standard deviation s of shear strength of the adhesive). This attempts to bracket the endurance limit $\tau_D(N_F,\ \tau_m)$ by a succession of failures and non-failures.

A.2 Method

An uneven number of specimens is tested.

The jth specimen is tested at the stress amplitude τ_{aj} close to the assumed $\tau_D(N_F)$. If failure occurs, the next specimen should be tested at $\tau_{aj+1} = \tau_{aj} - d$. If there is no failure the next specimen should be tested at $\tau_{aj+1} = \tau_{aj} + d$ until all the specimens have been used.

The stresses should be numbered from the lowest amplitude tested, which is denoted by the index $i = 0$, i.e. τ_a^0. The endurance limit is given by the equation

$$\tau_D(N_F,\ \tau_m) = \tau_a^0 + d\left[\frac{A}{L} \pm \left(\frac{1}{2}\right)\right]$$

where

τ_a^0 is the amplitude of the smallest stress used during the test;

d is the space between two amplitudes;

L is the number of least frequent possibilities (failure or non-failure) for the whole of the test sequence;

$$A = \sum_{i=0}^{i=k} i \times n_i$$

in which

n_i is the number of times that the least frequent possibility is observed at level of index i;

k is the number of levels necessary to pass from a certain failure event to a certain non-failure event $(0 < i < k)$.

The value $-(1/2)$ is used if failure is the least frequent; $+(1/2)$ is used if non-failure is the least frequent.

A.3 Calculation of the standard deviation of the limit of endurance

The estimation of the standard deviation is calculated by the formula:

$$s = 1{,}62 \times d\left(\frac{L \times B - A^2}{L^2} + 0{,}029\right)$$

with

$$B = \sum_{i=0}^{i=k} i^2 \times n_i$$

providing that

$$\frac{L \times B - A^2}{L^2} > 0{,}3$$

A.4 Typical example of staircase method

Assuming that:

$N_F = 10^6$

$\tau_m = 10$ MPa

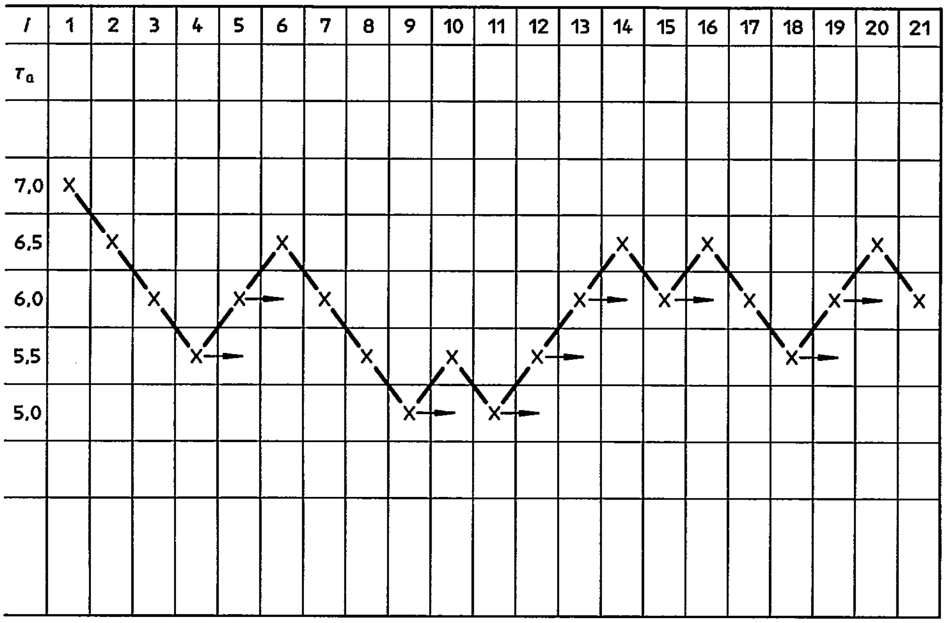
$\tau_D(10^6,\ 10) = 5{,}86$ MPa

$s = 0{,}52$ MPa

$$\tau_D(N_F,\ \tau_m) = \tau_a^0 + d\left[\frac{A}{L} \pm \left(\frac{1}{2}\right)\right]$$

For 21 specimens, an endurance limit of 5,86 MPa is obtained and a standard deviation of 0,52 MPa.

The same calculation carried out on the first 11 specimens would lead to an endurance limit of 5,62 MPa, and for the first seven specimens, to 6,00 MPa.



τ_a' MPa	i	n_i	in_i	i^2n_i
7,00	4	0	0	0
6,50	3	0	0	0
6,00	2	4	8	16
5,50	1	3	3	3
5,00	0	2	0	0
		$L=9$	$A=11$	$B=19$

Staircase method for 21 specimens { failed 12 (X) / not failed 9 (X ——►)

Figure A.1 — Example of the staircase method

Annex B
(normative)

Data reclassification method

B.1 Method of surfaces

A particular version of this method is known as the "data reclassification method". It is useful for obtaining a fairly precise estimate of the endurance limit without having to assume that the endurance limit follows a normal rate. The analogy between the formula giving the endurance limit and that supplied by the staircase method shall be noted.

B.2 Principle

The particular case of the general version should be used, where the test-stress amplitudes τ_{al}, τ_{ai}, τ_{ak} are regularly spaced by a step d, with the same number of specimens tested at each level. The endurance limit is provided by the equation:

$$\tau_D(N_F, \tau_m) = \tau_{ak} - d\left(\frac{T}{q} - \frac{1}{2}\right)$$

where

T is the total number of specimens failed before the number of fault test cycles N_F;

q is the number of specimens tested at each level i;

τ_{ak} is the highest stress amplitude tested having consistently led to failure.

B.3 Calculation of standard deviation of the endurance limit

Using the above notation, estimate the standard deviation for $\tau_D(N_F)$ using:

$$s = \left[d^2 \sum_{i=2}^{i=k-1} \frac{P_i(1-P_i)}{(q-1)}\right]^{\frac{1}{2}}$$

where P_i is the proportion of specimens failed at stress amplitude τ_{ai}.

B.4 Example of method of surfaces

Particular cases of equidistant levels with equal numbers. Technical characteristics of specimens:

Specimens made of steel bonded with a typical one-component epoxy adhesive.

16 specimens were selected at random from a batch of laboratory manufacture [see figure 4 a)].

Observations eliminated: None.

Table B.1 — Example of data

Test data:	Test fault cycles N_F = 10^6 cycles Step d = 0,2 MPa Number = 4 per level τ_m = 13 MPa		
Test index (i)	**Stress amplitude** (τ_{ai}) MPa	**Number of failures**	P_i
4	3,5	4	1
3	3,3	1	0,25
2	3,1	3	0,75
1	2,9	0	0

Number of specimens tested per level: $q = 4$

Total number of specimens failed before the number of fault test cycles N_F: $T = 8$

Proportion of specimens failed: $T/q = 2$

Calculation of limit of endurance $\tau_D(N_F, \tau_m)$:

$$\tau_{a4} - d\left(\frac{T}{q} - \frac{1}{2}\right) = 3,2 \text{ MPa}$$

i.e.

$$\tau_D(10^6, 13) = 3,2 \text{ MPa}$$

and

$$s = 0,07 \text{ MPa}$$

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