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Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies —

Part 4: Environmental conditions for durability

Plastiques — Évaluation des performances de l'interface d'adhérence dans les assemblages plastique-métal —

Partie 4: Conditions environnementales pour la durabilité

Reference number
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Foreword

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The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

ISO 19095 consists of the following parts, under the general title *Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies*:

— *Part 1: Guidelines for the approach*

— *Part 2: Test specimens*

— *Part 3: Test methods*

— *Part 4: Environmental conditions for durability*

Introduction

Structures of heterogeneous materials are being manufactured in the automobiles and aerospace industry sectors where higher safety margins are required.

The existing test methods are not appropriate because the evaluation of the adhesive interface is difficult as the polymer material has a relatively low mechanical strength and therefore fractures outside the joints. Therefore, it is necessary to develop a methodology for the evaluation of the adhesive interfaces.

A test method to evaluate accurately the adhesion interface performance or standardization of long-term evaluation under harsh environments is also necessary.

The method in ISO 19095 is intended to ensure that the integrity of the joint is realized through the interface and that traceability of the value improves the data comparison.

This part of ISO 19095 defines the conditions to evaluate the long-term durability.

Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies —

Part 4: Environmental conditions for durability

SAFETY STATEMENT — Persons using this part of ISO 19095 should be familiar with normal laboratory practice, if applicable. This part of ISO 19095 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 conditions. It is recognized that some of the materials permitted in this part of ISO 19095 might have a negative environmental impact. As technological advances lead to more acceptable alternatives for such materials, they will be eliminated to the greatest extent possible. At the end of the test, care should be taken to dispose of all waste in an appropriate manner in accordance with local regulations.

1 Scope

This part of ISO 19095 specifies the environmental conditions to evaluate the durability for the adhesion interface performance in plastic-metal assemblies.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

ISO 19095-1:2015, *Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies —Part 1: Guideline for the approach*

ISO 19095-2, *Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies — Part 2: Test specimens*

ISO 19095-3, *Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies — Part 3: Test methods*

IEC 60068-2-11, *Basic environmental testing procedures — Part 2-11: Tests — Test Ka: Salt mist*

3 Terms and definitions

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

3.1
shear stress
τ
<fatigue test> stress determined by dividing the force by the bonded surface area

Note 1 to entry: It is expressed in megapascals (MPa).

3.2
static shear stress

τ_R

<fatigue test> average static shear stress at rupture as determined by ISO 4587

Note 1 to entry: It is expressed in megapascals (MPa).

3.3
stress cycle

<fatigue test> smallest part of the stress/time function which is repeated at regular intervals

Note 1 to entry: It is of sinusoidal form (see Figure 1) with undulating shear.

Note 2 to entry: Cyclic stress can be considered to be the superposition of an alternating stress on a static stress which is the mean stress.

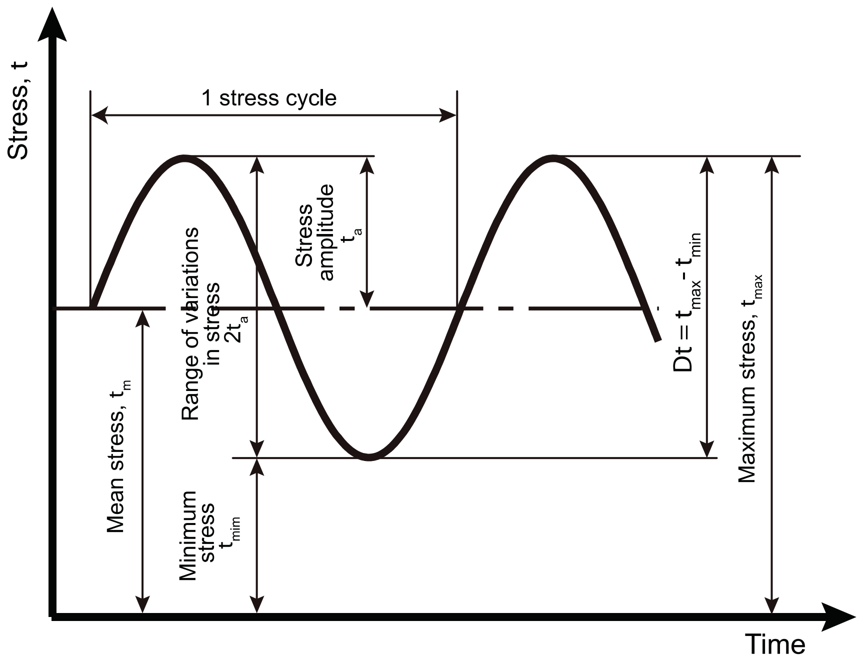


Figure 1 — Fatigue stress cycle

3.4
maximum stress

τ_{max}

<fatigue test> greatest algebraic value reached at regular intervals by the stress

Note 1 to entry: It is expressed in megapascals (MPa).

3.5
minimum stress

τ_{min}

<fatigue test> smallest algebraic value reached at regular intervals by the stress

Note 1 to entry: This stress shall always be positive and is expressed in megapascals (MPa).

3.6
mean stress

τ_m

<fatigue test> algebraic mean of the maximum and minimum stresses

Note 1 to entry: It is expressed as:

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

3.7
stress amplitude
τ_a
<fatigue test> alternating stress equal to half the algebraic difference between the maximum and minimum stresses

Note 1 to entry: It is expressed in megapascals (MPa).

Note 2 to entry: It is expressed as:

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

3.8
fatigue limit
τ_D
<fatigue test> limiting value which the stress amplitude τ_a approaches when the number of cycles becomes very large, for a given mean stress τ_m

Note 1 to entry: 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.

3.9
limit of endurance
$\tau_D(N_F)$
<fatigue test> shear stress determined at a specific number of fault test cycles N_F

Note 1 to entry: It is expressed in megapascals (MPa).

Note 2 to entry: The tests are carried out at constant τ_m, and the results should be presented in the form:

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

3.10
service life
N
<fatigue test> number of stress cycles applied to a specimen until it has reached the chosen end of the test

Note 1 to entry: Where it has not failed, the service life is not defined but is termed greater than the test duration.

3.11
cycle ratio
n/N
<fatigue test> ratio of the number of applied cycles (n) to the service life (N)

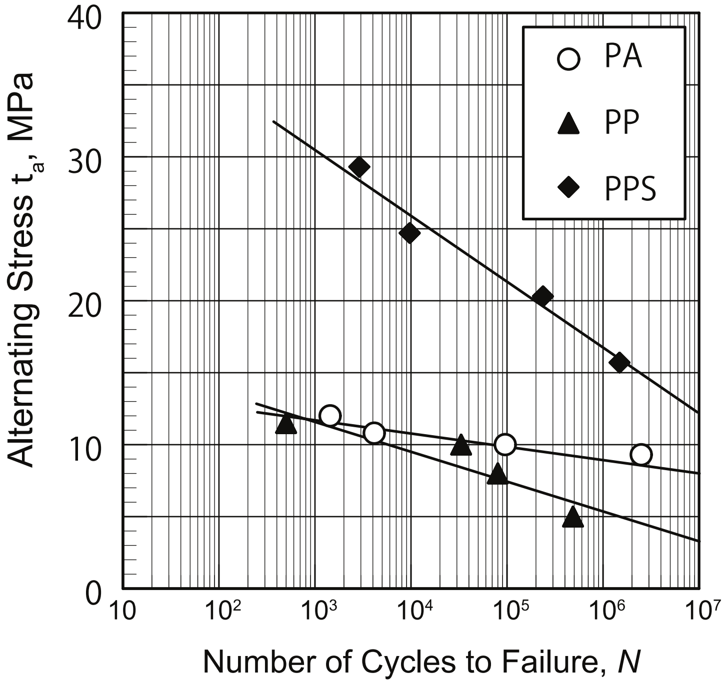
Note 1 to entry: This ratio is used in tests with load bearings, together with an SN curve (Woehler's curve).

3.12
SN curve
<fatigue test> 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 1)

Note 1 to entry: This curve is established by keeping τ_m constant. The SN curve is defined by the relationship between amplitude of stress and service life. On this curve (Figure 2), we can distinguish

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

—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.



Key

PA polyamide

PP polypropylene

PPS polyphenylene sulfide

Figure 2 — Semi-logarithmic plots of typical SN curves of plastic-aluminium 5052 assemblies tested at 30 Hz at room temperature

4 Test specimen

4.1 Form of test specimen

See ISO 19095-2.

4.2 Condition of test specimen

See ISO 19095-3.

5 Test procedure

5.1 Temperature dependence test

5.1.1 Apparatus

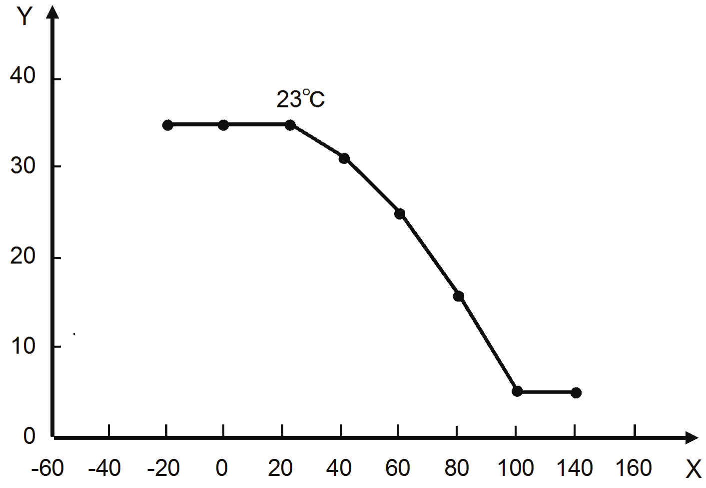
See ISO 19095-1.

5.1.2 Test procedure

After keeping the test specimen under the condition described in ISO 19095-1 for 48 h, select the corresponding temperature groups from (−40 ± 3) °C, (−20 ± 3) °C, (0 ± 2) °C, (40 ± 2) °C, (60 ± 2) °C, (80 ± 2) °C, (100 ± 2) °C, (120 ± 3) °C, (140 ± 2) °C, (160 ± 3) °C, and (180 ± 2) °C and carry out the test. The test specimen shall be kept at the specified temperature for 10 min.

5.1.3 Presentation of result

Based on the strength in respective temperature obtained in 5.2, develop a strength-temperature line graph. Figure 3 shows an example.



Key

X temperature (°C)

Y strength (MPa)

Figure 3 — Sample line graph of strength-temperature

5.2 Thermal shock test

5.2.1 Apparatus

See ISO 19095-1.

5.2.2 Test procedure

After keeping the test specimen under the condition described in ISO 19095-1 for 48 h, leave the test specimen in a constant low temperature chamber preset to (−40 ± 3) °C for (2 ± 0,5) h.

Take the test specimen out from the chamber and place it immediately in the chamber adjusted to the high temperature shown in Table 1, and repeat the temperature cycle as specified in Table 1 and Figure 4.

Respectively, after 10, 50, 100, 200, 500, 1 000 repetitions of the temperature cycle, take the test specimen out from the chamber, leave it under the condition described in ISO 19095-1 for more than 2 h, and carry out the test.

Table 1 — Conditions of thermal shock test

Classification	Preset temperature		Exposure time	
	High	Low	T2, T4	T1, T3
I	80 °C	−40 °C	1 h	5 min or less
II	120 °C			
III	150 °C			

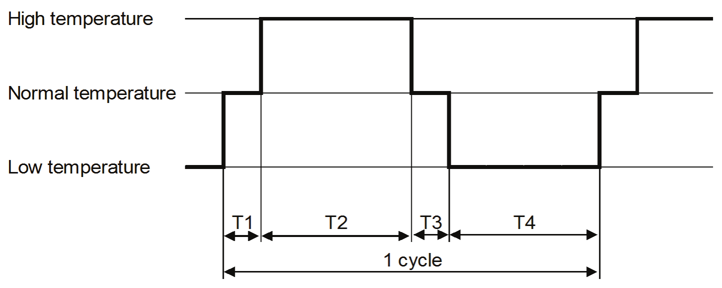


Figure 4 — Thermal shock test pattern

5.2.3 Presentation of result

Based on the performance result after each number of repetitions obtained in 5.1.2, develop a strength-repetitions line graph.

5.3 Temperature/Humidity cyclic test

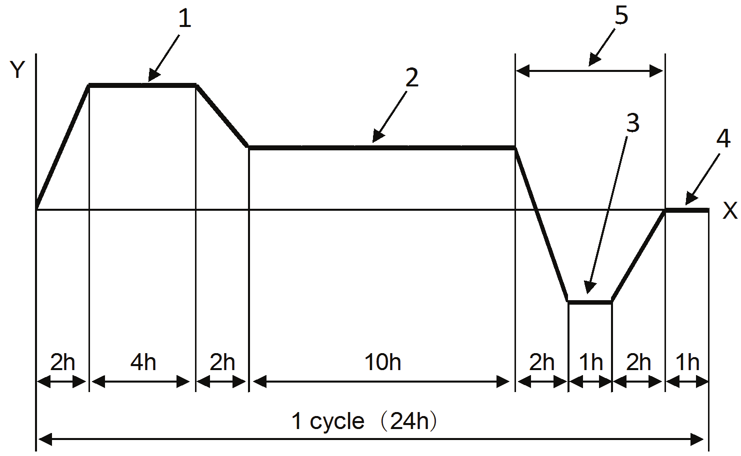
5.3.1 Apparatus

See ISO 19095-1.

5.3.2 Test procedure

After keeping the test specimen under the condition described in ISO 19095-1 for 48 h, place the piece in a constant temperature and humidity chamber and set the chamber to (25 ± 2) °C and (65 ± 20) % of humidity. Leave it under such condition for (2,5 ± 0,5) h and conduct the test in accordance with the patterns specified in Figure 5.

Respectively, after 2, 5, 10, 20 repetitions of each pattern, take the test piece out from the chamber, leave it under the condition for between 1 h and 2 h as described in ISO 19095-1, and carry out the test.



Key

X time

Y temperature (°C)

1 (60 ± 2) °C, (90 ± 5) % RH

2 45 ± 2) °C, (95 ± 5) % RH

3 (−10 ± 2) °C

4 (23 ± 2) °C, (65 ± 2) % RH

5 interval without humidity control

Figure 5 — Temperature/Humidity cyclic test pattern

5.3.3 Presentation of result

Based on the performance result after each number of repetitions obtained in 5.3.2, develop a strength-repetitions line graph.

5.4 High temperature and high humidity test

5.4.1 Apparatus

See ISO 19095-1.

5.4.2 Test procedure

After keeping the test specimen under the condition for 48 h as described in ISO 19095-1, carry out the test in accordance with the conditions specified in Table 2.

After leaving a test specimen in a chamber respectively for 24 h, 48 h, 168 h, 336 h, 672 h, 1 000 h, 1 500 h, 2 000 h of testing time, take the test specimen out from the chamber, leave it for more than 2 h under the condition described in ISO 19095-1:2015, Clause 4, and carry out the test.

Table 2 — Conditions of high temperature and high humidity test

Test temperature	(85 ± 2)°C
Relative humidity	(85 ± 2) %

5.4.3 Presentation of result

Based on the performance result for each testing time obtained in 5.4.2, develop a strength-testing time line graph.

5.5 Salt mist test

5.5.1 Apparatus

See ISO 19095-1.

5.5.2 Test procedure

After keeping the test piece under the condition for 48 h as described in ISO 19095-1:2015, Clause 4, carry out the test in accordance with the provisions of IEC 60068-2-11, Clause 4 to Clause 7.

After leaving a test piece in a chamber respectively for 24 h, 48 h, 96 h, 168 h, 336 h, 672 h, 1 000 h, 1 500 h, 2 000 h of testing time, take the test piece out from the chamber, and clean for 5 min with running tap water. Subsequently, clean it with distilled water or deionization water and remove dripping using an air-spray. Here, the temperature of water used for cleaning shall be 35 °C or lower.

After removing the drips, leave the test piece for between 1 h and 2 h under the condition described in ISO 19095-1:2015, Clause 4 and carry out the test.

5.5.3 Presentation of result

Based on the outcome of 5.5.2, develop a strength-test time graph.

5.6 Fatigue test

5.6.1 Principle

The specimen is cyclically stressed in a way that can 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.

5.6.2 General conditions

Plastic-metal assemblies can 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, 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.

5.6.3 Apparatus

See ISO 19095-1.

5.6.4 Test procedure

After keeping the test specimen under the condition for 48 h described in ISO 19095-1, unless otherwise specified separately, carry out the test in accordance with the condition specified in Table 3 referring as follows.

Table 3 — Fatigue test conditions

Frequency	30 Hz
Mean stress (τ_m)	0,35 τ_R

5.6.4.1 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.

5.6.4.1.1 Test at least four specimens for each of the amplitudes τ_a chosen, such that, the failures occur between 10^4 and 10^7 cycles. A minimum of three different amplitudes is necessary in this range. Determine the limit of endurance at 10^7 fault test cycles N_F.

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 τ_d (N_F, τ_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).

5.6.4.1.2 Plot the SN curve, passing through the centre-line points and the endurance limit τ_δ (N_F, τ_m) with coordinates (τ_a, log N) which permits a straight line to be obtained.

5.7 Report

In addition to the report described in ISO 19095-2, the following shall be included in the test report as appropriate:

a) number of this part of ISO 19095, i.e. ISO 19095-4;

b) pre-processing conditions;

c) type of environment, conditions, and the number of repetitions (time) of the test;

d) form of test specimen;

e) test result;

f) test date.

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 $\tau_{\mathrm{d}}(N_{\mathrm{F}},\tau_{\mathrm{m}})$ 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 $\tau_{\mathrm{D}}(N_{\mathrm{F}})$. 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 Formula (A.1).

$$\tau_{\mathrm{d}}(N_{\mathrm{F}},\tau_{\mathrm{m}})=\tau_a^0+d\left(\frac{A}{L}-\frac{1}{2}\right) \tag{A.1}$$

where

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

$$A=\sum_{i=0}^{i=k} i\times n_i \tag{A.2}$$

where

n_i	is the number of times that the least frequent possibility is observed at level of index i;
k	is the number of level 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 standard deviation of the limit of endurance

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

$$s = 1{,}62 \times d \left(\frac{L \times B - A^2}{L^2} + 0{,}029 \right) \tag{A.3}$$

with

$$B = \sum_{i=0}^{i=k} i^2 \times n_i \tag{A.4}$$

providing that

$$\frac{L \times B - A^2}{L^2} > 0{,}3 \tag{A.5}$$

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

$$\tau_d(N_F, \tau_m) = \tau_a^0 + d\left[\frac{A}{L} \pm \left(\frac{1}{2}\right)\right] \tag{A.6}$$

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.

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 Formula (B.1):

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

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:

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

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.

Observations eliminated: None.

Table B.1 — Example of data

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
Test data:	Test fault cycles Step Number	$N_F = 10^6$ cycles d = 0,2 MPa = 4 per level τ_m = 13 MPa	

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

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

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

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

$$\tau_{a^4} - d\left(\frac{T}{q} - \frac{1}{2}\right) = 3{,}2 \text{ MPa} \quad \text{(B.3)}$$

i.e.

$$\tau_D(10^6,\ 13) = 3{,}2 \text{ MPa} \quad \text{(B.4)}$$

and

$$s = 0{,}07 \text{ MPa} \quad \text{(B.5)}$$

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