
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Mechanical properties of ceramic
composites at high temperature
in air at atmospheric pressure —
Determination of fatigue properties at
constant amplitude**

*Céramiques techniques — Propriétés mécaniques des composites
céramiques à haute température sous air à pression atmosphérique
— Détermination des propriétés de fatigue à amplitude constante*



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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at high temperature in air at atmospheric pressure — Determination of fatigue properties at constant amplitude

1 Scope

This International Standard specifies the conditions for the determination of properties at constant-amplitude of load or strain in uniaxial tension/tension or in uniaxial tension/compression cyclic fatigue of ceramic matrix composite materials (CMCs) with fibre reinforcement for temperature up to 1 700 °C in air at atmospheric pressure.

This International Standard applies to all ceramic matrix composites with fibre reinforcement, unidirectional (1D), bi-directional (2D), and tri-directional (xD, where $2 < x \leq 3$).

The purpose of this International Standard is to determine the behaviour of CMC when subjected to mechanical fatigue and oxidation simultaneously. Tests for the determination of fatigue properties at high temperature in inert atmospheres differ from those in oxidative atmospheres. Contrary to an inert atmosphere, damage in an oxidative atmosphere accumulates due to the influence of purely mechanical fatigue and to chemical effects of the material's oxidation.

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 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 9513, *Metallic materials — Calibration of extensometer systems used in uniaxial testing*

ISO 4574, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at high temperature — Determination of compression properties*

ISO 14574, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at high temperature — Determination of tensile properties*

ISO 15733, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of ceramic composites at ambient temperature in air atmospheric pressure — Determination of tensile properties*

IEC 60584-1, *Thermocouples — Part 1: EMF specifications and tolerances*

IEC 60584-2, *Thermocouples — Part 2: Tolerances*

CEN/TR 13233, *Advanced technical ceramics — Notations and symbols*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TR 13233¹⁾ and the following apply.

3.1 General

3.1.1 test temperature

T

temperature of the test specimen at the centre of the gauge length

3.1.2 calibrated length

l

part of the test specimen which has uniform and minimum cross-section area

3.1.3 gauge length

L_0

initial distance between reference points on the test specimen in the calibrated length

3.1.4 controlled temperature zone

part of the calibrated length including the gauge length where the temperature is within 50 °C of the test temperature

3.1.5 initial cross-section area

S_0

initial cross-section area of the test specimen within the calibrated length, at the test temperature

Note 1 to entry: Two initial cross-section areas of the test specimen can be defined:

- apparent cross-section area: this is the total area of the cross-section, $S_{0\text{ app}}$;
- effective cross-section area: this is the total area corrected by a factor to account for the presence of a coating, $S_{0\text{ eff}}$.

3.1.6 longitudinal deformation

A

change in the gauge length between reference points under an uniaxial force

3.1.7 strain

ε

relative change in the gauge length defined as the ratio, A/L_0

3.1.8 stress

σ

force supported by the test specimen at any time in the test, divided by the initial cross-section area

Note 1 to entry: Two stresses can be distinguished:

- apparent stress, σ_{app} , when the apparent cross-section area (or total cross-section area) is used;
- effective stress, σ_{eff} , when the effective cross-section area is used.

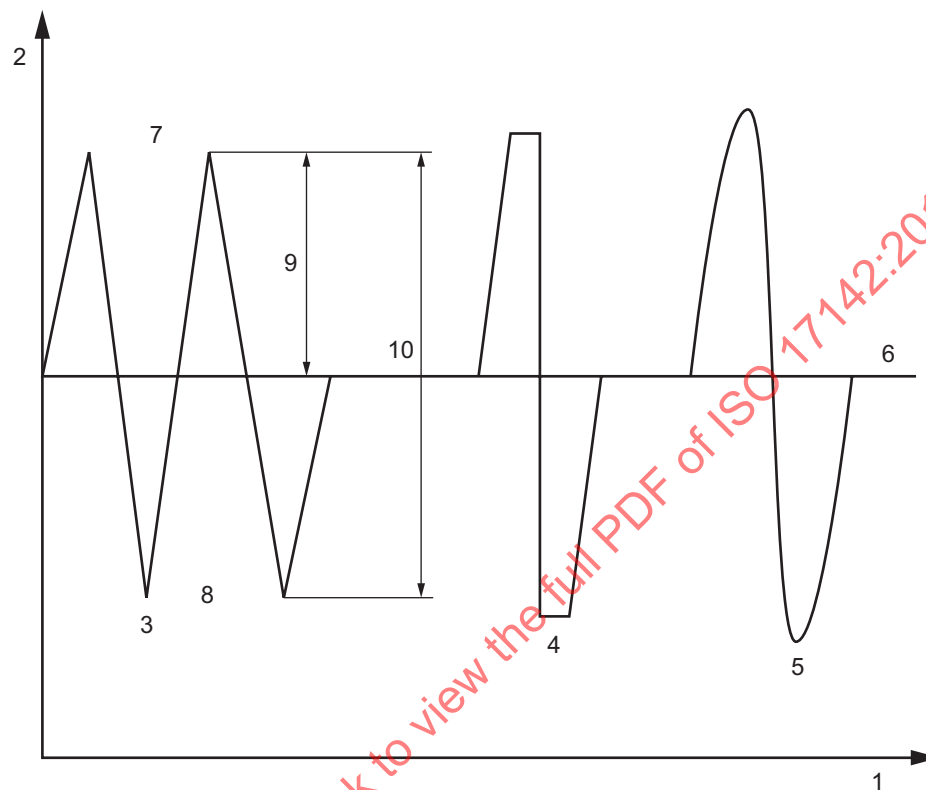
1) Intended to be substituted by a future International Standard.

Note 2 to entry: Stress can be either in tension or in compression.

3.1.9

constant amplitude loading

in cyclic fatigue loading, constant wave form loading in which the peak loads and the valley loads are kept constant during the test (see [Figure 1](#) for nomenclature relevant to cyclic fatigue testing)



Key

1	time	6	mean
2	control parameter (test mode)	7	peak (maximum)
3	triangular form	8	valley (minimum)
4	trapezoidal form	9	amplitude
5	sinusoidal form	10	range

Figure 1 — Cyclic fatigue nomenclature and wave forms

3.2 Cyclic fatigue phenomena

NOTE Stress-strain curve parameters are defined as given in [Figure 2](#).

3.2.1 Load ratio

3.2.1.1

load ratio

R

in cyclic fatigue loading, algebraic ratio of the two loading parameters of a cycle

Note 1 to entry: The most widely used ratios are

- $R = (\text{minimum load}/\text{maximum load})$, or
- $R = (\text{valley load}/\text{peak load})$.

3.2.2 Cyclic fatigue stress

3.2.2.1

maximum stress

$\sigma_{\max}$

maximum applied stress during cyclic fatigue

3.2.2.2

minimum stress

$\sigma_{\min}$

minimum applied stress during cyclic fatigue

3.2.2.3

mean stress

σ_m

average applied stress during cyclic fatigue

Note 1 to entry: $\sigma_m = \frac{\sigma_{\max} + \sigma_{\min}}{2}$

3.2.2.4

stress amplitude

σ_a

difference between the maximum stress and the minimum stress

Note 1 to entry: $\sigma_a = \frac{\sigma_{\max} - \sigma_{\min}}{2} = \sigma_{\max} - \sigma_m = \sigma_m - \sigma_{\min}$

3.2.3 Cyclic fatigue strain

3.2.3.1

maximum strain

$\epsilon_{\max}$

maximum applied strain during cyclic fatigue

3.2.3.2

minimum strain

$\epsilon_{\min}$

minimum applied strain during cyclic fatigue

3.2.3.3

mean strain

ϵ_m

average applied strain during cyclic fatigue

Note 1 to entry: $\epsilon_m = \frac{\epsilon_{\max} + \epsilon_{\min}}{2}$

3.2.3.4

strain amplitude

ϵ_a

difference between the maximum stress and the minimum stress

Note 1 to entry: $\epsilon_a = \frac{\epsilon_{\max} - \epsilon_{\min}}{2} = \epsilon_{\max} - \epsilon_m = \epsilon_m - \epsilon_{\min}$

3.2.4 Fatigue parameters

3.2.4.1

number of cycles

N

total number of loading cycles which is applied to the test specimen during the test

3.2.4.2**cyclic fatigue life** N_f

total number of loading cycles which is applied to the test specimen up to failure

3.2.4.3**time to failure** t_f

time duration required to obtain the number of cycles, N_f

4 Principle

A test specimen of specified dimensions is heated to the testing temperature and tested in cyclic fatigue as follows:

- method A: the test specimen is cycled between two constant stress levels at a specified frequency;
- method B: the test specimen is cycled between two constant strain levels at a specified frequency.

The total number of cycles is recorded. If strain is not determined, only the lifetime duration or the residual mechanical properties can be determined. If strain is determined, a number of stress-strain cycles are recorded at specified intervals to determine damage parameters, in addition to the lifetime duration and residual mechanical properties.

NOTE Residual properties can be determined on the test specimens which have not failed during the test using the methods described in the appropriate International Standards.

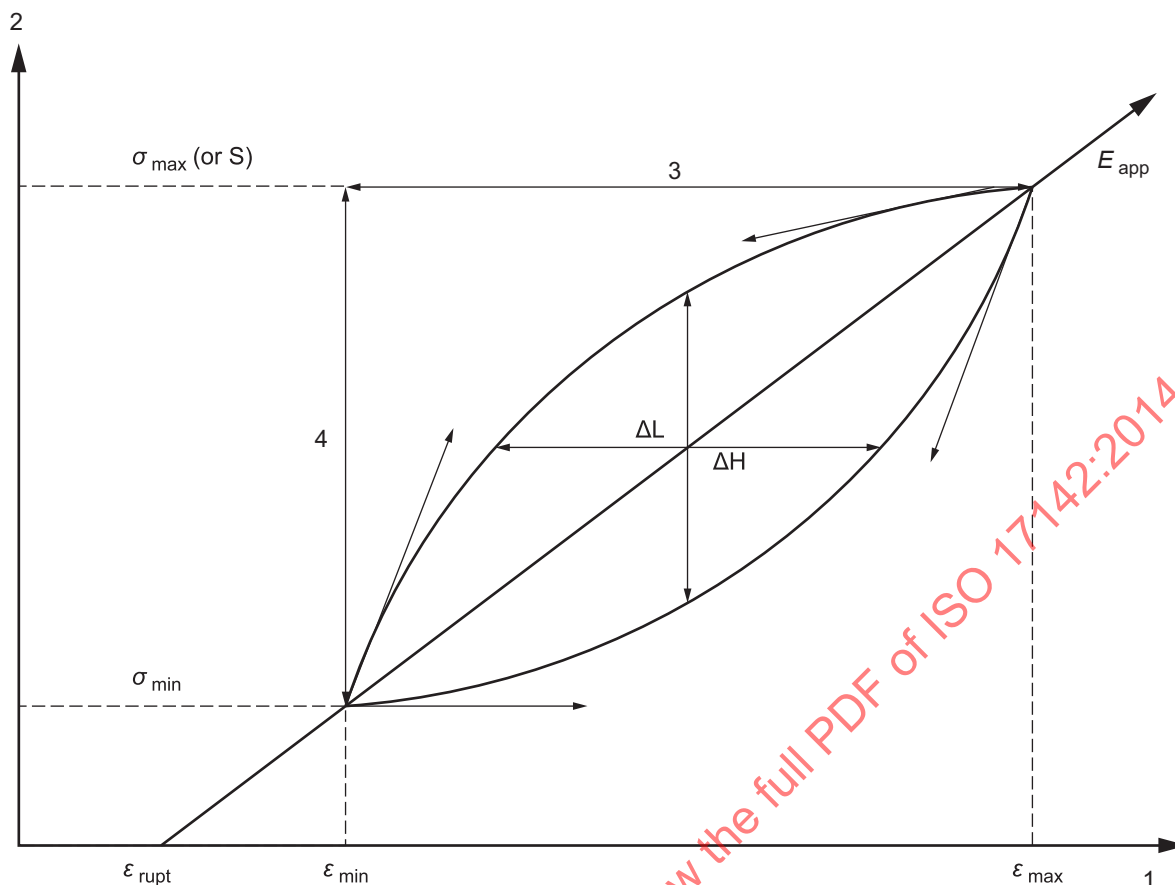
5 Significance and use

This test method enables characterization of the cyclic fatigue behaviour at constant amplitude of CMCs subjected to long duration loading. The simplest way to determine the fatigue properties of a material is to establish lifetime diagrams. In these diagrams, the time to failure (or the cyclic fatigue life) is plotted versus stress (or strain) amplitude.

The complete lifetime diagram requires the use of a great number of test specimens, which is expensive and time consuming. Hence, it is sufficient to know the cyclic-fatigue under specified stress (or strain) conditions, or to measure the fatigue limit. In any case, the typical fatigue test is defined by cyclic loading, constant amplitude, environment, temperature, and frequency.

To better characterize the mechanical behaviour during a fatigue test, it is possible to determine several mechanical parameters from stress-strain curves. These parameters can then be plotted versus time or versus number of cycles. This displays the damage evolution during the cyclic loading. The following parameters can be considered (see [Figure 2](#)):

- the residual strain at zero load;
- the secant elastic modulus, or the relative damage parameters;
- the area of the stress-strain hysteresis loop, or the internal friction;
- the maximum strain, the minimum strain, or the difference between them for a selected cycle;
- some specific tangent elastic moduli, for example at the top or at the bottom of the stress-strain loop.



Key

- 1 strain (ϵ)
- 2 stress (σ)
- 3 width (L)
- 4 height (H)

Figure 2 — Parameters that can be considered to assess the cyclic fatigue behaviour

6 Apparatus

6.1 Fatigue test machine

A hydraulic type or electric actuator driven test machine shall be used. It shall be load or strain control operated.

The system for measuring the force applied to the test specimen shall be specially designed for fatigue tests and shall conform to grade 1 or better, in accordance with ISO 7500-1. This shall apply during actual test conditions. The machine shall be equipped with a cycle counter for the chosen test frequency.

6.2 Load train

The load train configuration shall ensure that the load indicated by the load cell and the load experienced by the test specimen are the same.

The load train performance including the alignment system and the force transmitting system shall not change because of heating.

The attachment fixtures shall align the test specimen axis with that of the applied force.

The grip design shall prevent the test specimen from slipping.

The use of hydraulic grips is recommended. In this case, the cooled grip technique where the grips are outside the hot zone, is used.

NOTE In this technique, a temperature gradient exists between the centre which is at the prescribed temperature and the ends which are at the same temperature as the grips.

6.3 Set-up for heating

The set-up for heating shall be constructed in such a way that the temperature in the controlled temperature zone remains within 50 °C of the test temperature.

6.4 Extensometer

If applicable, extensometry shall be capable of continuously recording the longitudinal deformation at test temperature and compatible with the chosen test frequency. The extensometer shall conform to class 1 or better, in accordance with ISO 9513.

The commonly used type of extensometer is the mechanical type.

In this case, the gauge length is the longitudinal distance between the two locations where the extensometer rods contact the test specimen. The rods may be exposed to temperatures higher than the test specimen temperature. Temperature and/or environment induced structural changes in the rod material shall not affect the accuracy of deformation measurement. The material used for the rods shall be compatible with the test specimen material.

Care should be taken to correct for changes in calibration of the extensometer which might occur as a result of operating under conditions different from those for calibration.

The extensometer performance shall not change because of the test duration.

Rod pressure onto the test specimen shall be the minimum necessary to prevent slipping of the extensometer rods.

6.5 Temperature measurement

For temperature measurement, either thermocouples conforming to IEC 60584-1 and IEC 60584-2 shall be used, or, where thermocouples not conforming to IEC 60584 or pyrometers are used, they shall be appropriately calibrated and the calibration data added to the test report.

6.6 Data recording system

A calibrated recorder may be used to record force-deformation curve. The use of a digital data recording system is recommended.

6.7 Micrometers

Micrometers used for the measurement of the dimensions of the test specimen shall conform to ISO 3611.

7 Test specimens

The lifetime of CMCs depends, among other factors, on oxidation for given levels of temperature and stress or strain. Therefore, the configuration of the test specimen shall be designed to obtain a rupture in the controlled temperature zone. For this purpose, a dog-bone test specimen shall be used as specified in [Figure 3](#) and [Table 1](#).

In addition, the choice of the test specimen geometry depends on the nature of the material and of the reinforcement structure.

The volume in the gauge length shall be representative of the material. The total length, l_t , depends on the furnace and the gripping system.

NOTE Generally, the total length is greater than 150 mm.

In case of 1D composites, dog bone specimens are not recommended. Specimens without shoulders should be used.

In the case of tensile-compressive fatigue tests, the test specimen configuration shall be chosen to avoid buckling failure, as defined in ISO 14544.

Within the gauge length, any temperature variation shall remain within 30 °C of the test temperature.

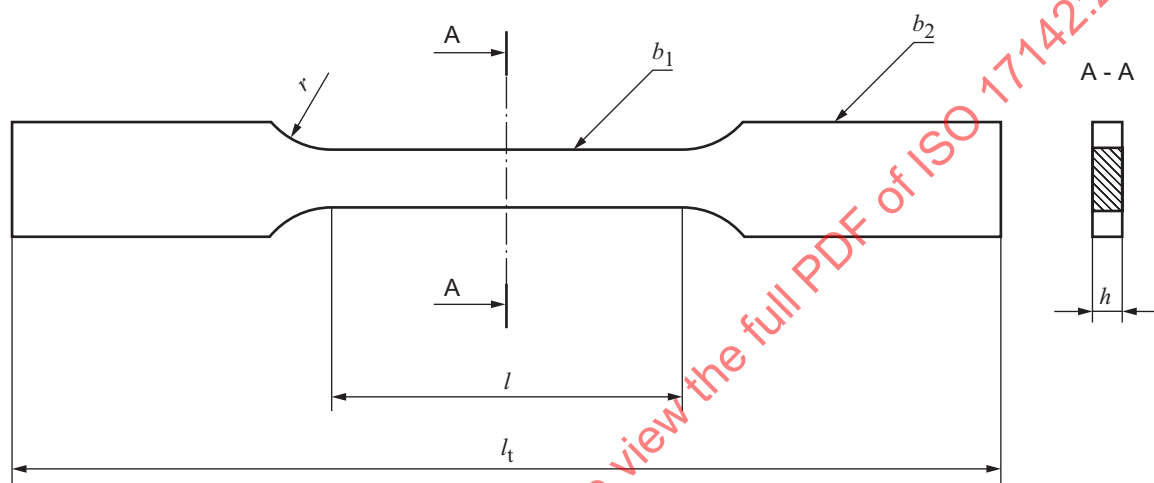


Figure 3 — Test specimen geometry for 2D or xD ($x > 2$) reinforcement

Table 1 — Recommended test specimen dimensions

Dimensions in millimetres

	1D, 2D, and xD	Tolerance
l , calibrated length	30 to 80	$\pm 0,5$
h , thickness	≥ 2	$\pm 0,2$
b_1 , width in the calibrated length ($x \geq 2$)	8 to 20	$\pm 0,2$
b_2 , width	$B_2 = \alpha b_1$ with $\alpha = 1,2$ to 2	$\pm 0,2$
r , radius ($x \geq 2$)	>30	± 2
Parallelism of machined parts	0,05	—

8 Test specimen preparation

8.1 Machining and preparation

During cutting out, care shall be taken to align the test specimen axis with the desired fibre related loading axis.

Machining parameters which avoid damage to the material shall be established and documented. These parameters shall be adhered to during test specimen preparation.

8.2 Number of test specimens

At least three valid test results, as specified in [9.4](#), are required for any condition.

9 Test procedure

9.1 Test set-up: temperature considerations

9.1.1 General

The following determinations shall be carried out under conditions representative of the tests and shall be repeated every time there is a change in material, in specimen geometry, in gripping configuration, etc. In establishing them, time shall be allowed for temperature stabilization.

9.1.2 Controlled temperature zone

Prior to testing, the temperature gradient within the calibrated length inside the furnace shall be established over the temperature range of interest. This shall be done by measuring the specimen temperature at a minimum of three locations, which shall be the two extensometer reference points and a point midway between the two.

To establish the length of the controlled temperature zone, it is necessary to measure the temperature also outside the gauge length. The temperature variation within the controlled temperature zone and the gauge length shall meet the requirements of [Clause 7](#) and [6.3](#) respectively.

Temperatures may be measured by any means conforming to [6.5](#). If thermocouples are used to measure the temperature at different locations of the specimen, they shall be embedded (and sealed if necessary) into the specimen to a depth approximately equal to half the specimen dimension in the direction of insertion.

9.1.3 Temperature calibration

During a series of tests, the test temperature can be determined either directly by measurement on the specimen itself, or indirectly from the temperature indicated by the temperature control device.

In the latter case, the relationship between the control temperature and test specimen temperature at the centre of the gauge length shall be established beforehand on a dummy test specimen over the range of temperature of interest.

NOTE The relationship between the temperature indicated by the temperature control system and the test temperature is usually established simultaneously with the controlled temperature zone.

9.2 Measurement of test specimen dimensions

The cross-section area shall be determined at the centre of the specimen and at each end of the gauge length.

The cross-section area varies with temperature and the variation is very difficult to measure. For this reason, the cross-section area shall be measured at room temperature.

Dimensions shall be measured to an accuracy of $\pm 0,01$ mm. The arithmetic means of the measurements shall be used for calculations.

9.3 Testing technique

9.3.1 Specimen mounting

Install the test specimen in the gripping system with its longitudinal axis coinciding with that of the test machine.

Care shall be taken not to induce flexural or torsional loads in the test specimen.

9.3.2 Setting the extensometer

In the case where a contacting extensometer is positioned at ambient temperature, the extensometer output shall be adjusted to read zero after the stabilization period at the test temperature.

In the case where the material has a high thermal expansion coefficient, it is recommended to mechanically pre-set the extensometer taking expansion into account in order to be close to zero when at test temperature.

9.3.3 Heating the test specimen

Raise the test specimen temperature to the required test temperature and maintain this temperature for a period to allow for temperature stabilization and, if applicable, for stabilization of the extensometer readout. Two procedures are possible: if test specimen temperature is measured during the test on the specimen itself, this temperature shall be used to control the furnace. If it is not possible to measure directly the test specimen temperature during the test, then it is necessary to use the relation between test specimen temperature and furnace temperature, which has been established in 9.1. Ensure that the test specimen stays in the initial state of stress during heating.

9.3.4 Measurements

- zero the load cell;
- zero the extensometer, if applicable;
- set the maximum number of cycles, N ;
- for method A, set the maximum and minimum stress values;
- for method B, set the maximum and minimum strain values;
- set the frequency and the wave shape;
- start the fatigue test:
 - for method A, in load control mode;
 - for method B, in strain control mode;
- record the number of cycles, N , or N_f ;
- if an extensometer is used, record the stress-strain loops up to the total number of cycles:

A specific computer program is recommended to control the test. Depending on the computerized facilities used, all the loops can be recorded. If this is not possible, the following sequence can be used to record stress versus strain:

- every cycle for the first 10 cycles;
- one cycle every 10 cycles between 10 and 100 cycles;
- one cycle every 100 cycles between 100 and 1 000 cycles;

- one cycle every 1 000 cycles between 1 000 and 10 000 cycles;
- etc.

9.4 Test validity

The following circumstances shall invalidate a test for the determination of the lifetime duration:

- failure to specify and record test conditions;
- specimen slippage in the grips;
- rupture in an area outside of the controlled temperature zone.

In addition, the following circumstances shall invalidate a test for the determination of the damage parameter:

- extensometer slippage;
- extensometer drift.

10 Calculation of results

10.1 Time to failure, t_f

Calculate the time to failure in accordance with the following formula:

$$t_f(\text{hours}) = \frac{N_f}{f \times 3\,600}$$

where

N_f is the number of cycles required to obtain failure of the test specimen;

f is the frequency, in hertz (Hz).

10.2 Damage parameters

Calculate the damage parameter, D_n , for each recorded cycle, n , in accordance with the following formula:

$$D_n = 1 - \frac{E_{n,\text{app}}}{E_{1,\text{app}}}$$

where

D_n is the damage parameter at the n^{th} fatigue cycle;

$E_{n,\text{app}}$ is the secant modulus at the n^{th} fatigue loop (see [Figure 4](#));

$E_{1,\text{app}}$ is the secant modulus at the first fatigue loop (see [Figure 4](#));

$E_{n,\text{app}}$ is equal to $\frac{\sigma_{\text{max}}}{\varepsilon_{\text{max}} - \varepsilon_{\text{nresidual}}}$ (see [Figure 4](#)).

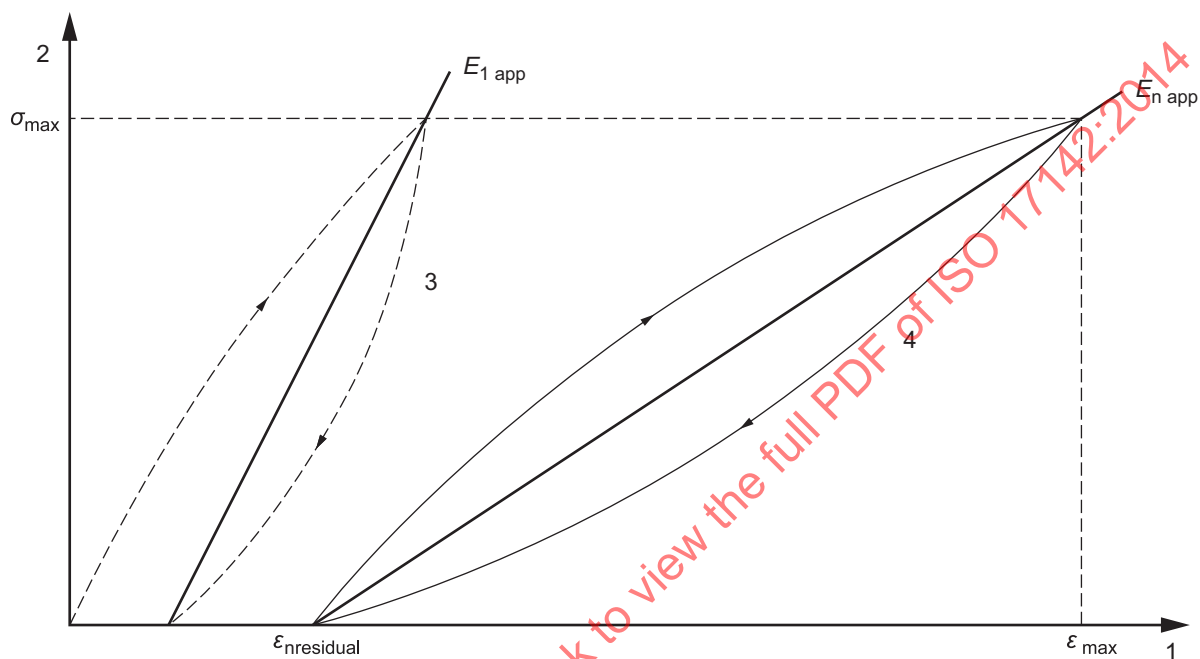
$\varepsilon_{\text{nresidual}}$ is equal to $\frac{\varepsilon_{\text{unl}} + \varepsilon_1}{2}$ [see [Figure 4 b](#)].

The damage parameters, D_n , $\varepsilon_{n\max}$, $\varepsilon_{n\text{residual}}$, can be plotted versus the number of cycles, N . For N , a log scale is almost always used, although a linear scale can also be used. Figures 4 a) to c) represent three cases of cyclic fatigue of CMCs.

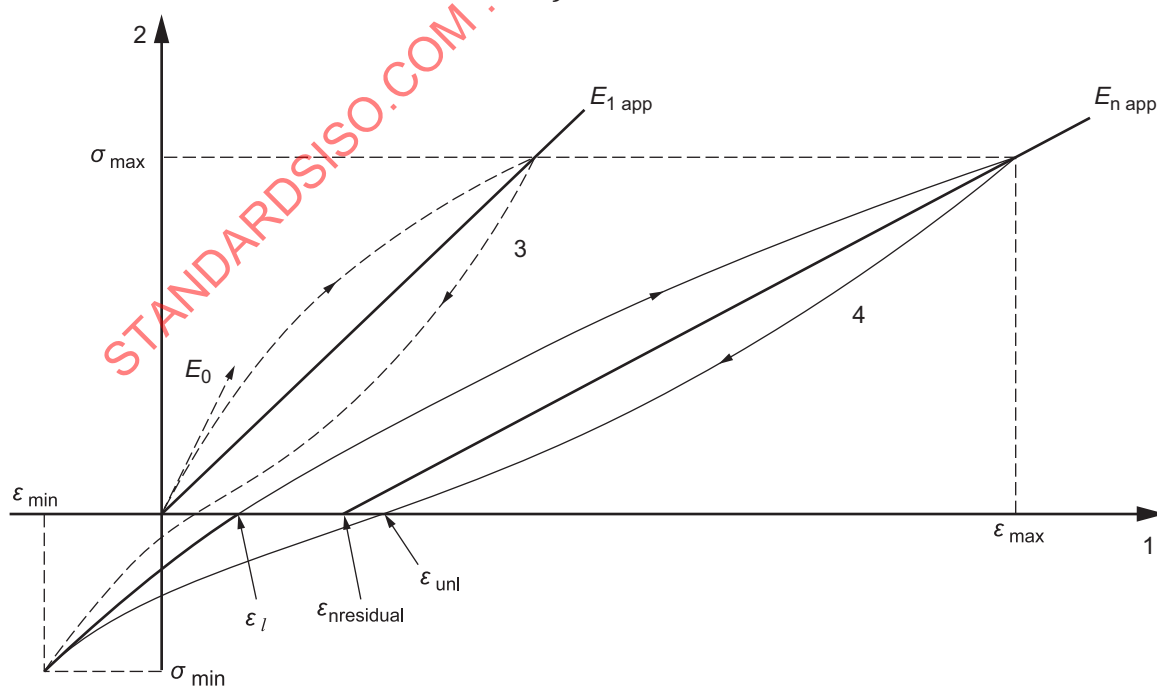
NOTE [Annex A](#) shows the schematic evolution of E under different circumstances of damage and creep.

10.3 Residual properties

When rupture does not occur before the end of the test, test specimen shall be tested to rupture in accordance with ISO 14574 or ISO 15733.



a) $R = 0$



b) $R < 0$