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**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**

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*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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

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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*.

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# 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 13233<sup>1)</sup> 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

$\varepsilon$

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

##### 3.1.8 stress

$\sigma$

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

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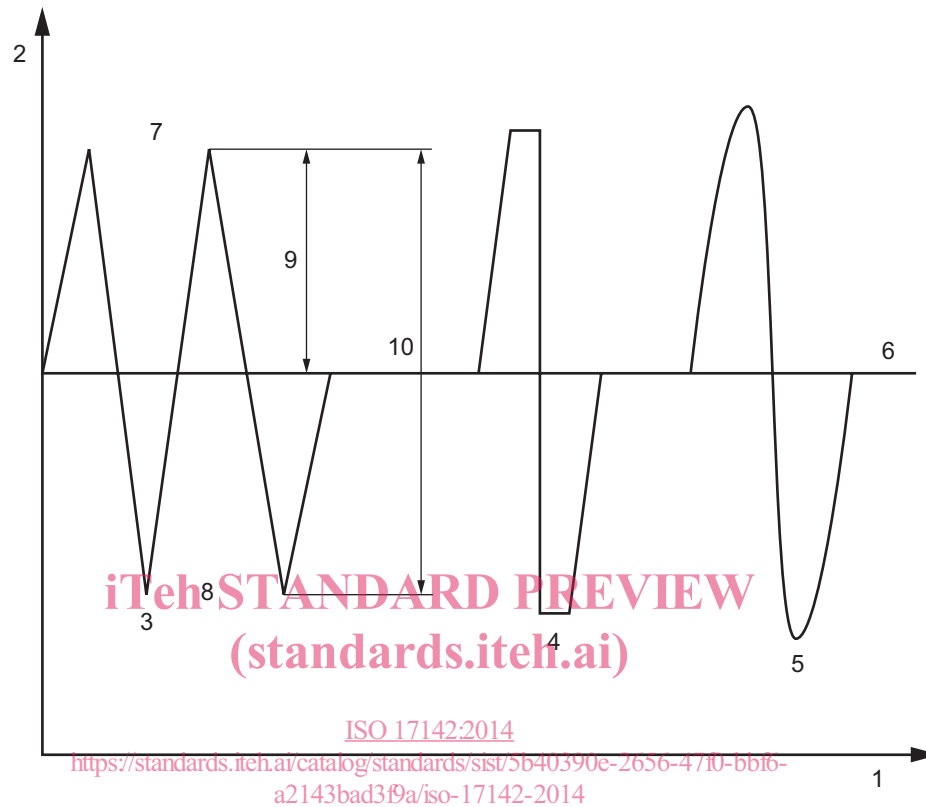
1) Intended to be substituted by a future International Standard.

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

$\sigma_m$   
average applied stress during cyclic fatigue

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

#### 3.2.2.4

##### stress amplitude

$\sigma_a$   
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

$\varepsilon_{\max}$   
maximum applied strain during cyclic fatigue

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#### 3.2.3.2

##### minimum strain

$\varepsilon_{\min}$   
minimum applied strain during cyclic fatigue

#### 3.2.3.3

##### mean strain

$\varepsilon_m$   
average applied strain during cyclic fatigue

Note 1 to entry:  $\varepsilon_m = \frac{\varepsilon_{\max} + \varepsilon_{\min}}{2}$

#### 3.2.3.4

##### strain amplitude

$\varepsilon_a$   
difference between the maximum stress and the minimum stress

Note 1 to entry:  $\varepsilon_a = \frac{\varepsilon_{\max} - \varepsilon_{\min}}{2} = \varepsilon_{\max} - \varepsilon_m = \varepsilon_m - \varepsilon_{\min}$

### 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.

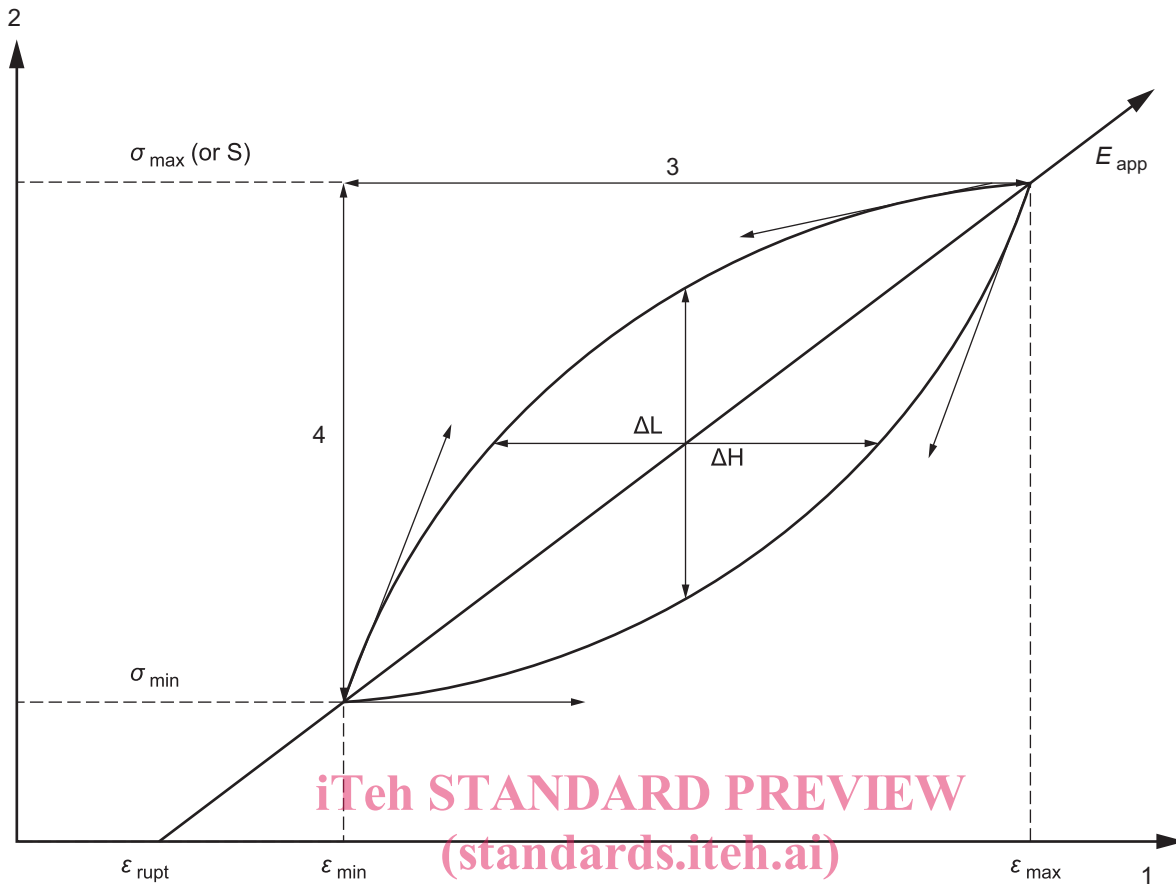
## 5 Significance and use

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 ( $\epsilon$ )
- 2 stress ( $\sigma$ )
- 3 width ( $L$ )
- 4 height ( $H$ )

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**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](#).