# INTERNATIONAL STANDARD

**ISO 204**

# Metallic materials — Uniaxial creep testing in tension — Method of test

*Matériaux métalliques — Essai de fluage uniaxial en traction — Méthode d'essai*

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# Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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.

ISO 204 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 1, *Uniaxial testing*.

This second edition cancels and replaces the first edition (ISO 204:1997), which has been technically revised.

# Introduction

This International Standard is an extensive revision of the first edition of ISO 204:1997 and incorporates many recommendations developed through the European Creep Collaborative Committee (ECCC).

New annexes have been added concerning temperature measurement using thermocouples and their calibration, creep testing test pieces with circumferential Vee and blunt (Bridgman) notches, estimation of measurement uncertainty and methods of extrapolation of creep rupture life.

NOTE      Information is sought relating to the influence of off-axis loading or bending on the creep properties of various materials. Consideration will be given at the next revision of this International Standard as to whether the maximum amount of bending should be specified and an appropriate calibration procedure be recommended. The decision will need to be based on the availability of quantitative data [39].

# Metallic materials — Uniaxial creep testing in tension — Method of test

## 1 Scope

This International Standard specifies the method for the uninterrupted and interrupted creep tests and defines the properties of metallic materials which can be determined from these tests, in particular the creep elongation and the time of creep rupture, at a specified temperature.

The stress rupture test is also covered by this International Standard, as is the testing of notched test pieces.

NOTE    In stress rupture testing, elongation is not generally recorded during the test, only the time to failure under a given load, or to note that a predetermined time was exceeded under a given force.

## 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 286-2, *ISO system of limits and fits — Part 2: Tables of standard tolerance grades and limit deviations for holes and shafts*

ISO 783 [1), *Metallic materials — Tensile testing at elevated temperature*

ISO 7500-2, *Metallic materials — Verification of static uniaxial testing machines — Part 2: Tension creep testing machines — Verification of the applied force*

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

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE    Several different gauge lengths and reference lengths are specified in this International Standard. These lengths reflect custom and practice used in different laboratories throughout the world. In some cases, the lengths are physically marked on the test piece as lines or ridges; in other cases, the length may be a virtual length based upon calculations to determine an appropriate length to be used for the determination of creep elongation. For some test pieces, $L_r$, $L_o$ and $L_e$ are the same length (see 3.1, 3.2 and 3.5).

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1)    To be revised by ISO 6892-2, *Metallic materials — Tensile testing — Part 2: Method of test at elevated temperature.*

**3.1**
**reference length**
$L_r$
base length used for the calculation of elongation

NOTE    A method to calculate this value is given in 7.5 for test pieces where the extensometer is attached to either ridges on the parallel length or to the shoulders of the test piece.

**3.1.1**
**original reference length**
$L_{ro}$
reference length determined at ambient temperature before the test

NOTE    In general, $L_{ro} \geqslant 5D$.

**3.1.2**
**final reference length**
$L_{ru}$
reference length determined at ambient temperature after rupture, with the pieces carefully fitted back together with their axes in a straight line

**3.2**
**original gauge length**
$L_o$
length between gauge length marks on the test piece measured at ambient temperature before the test

NOTE 1    In general, $L_o \geqslant 5D$.

NOTE 2    $L_o$ may also be used for the calculation of elongation.

**3.3**
**final gauge length after rupture**
$L_u$
length between gauge length marks on the test piece measured after rupture, at ambient temperature, with the pieces carefully fitted back together with their axes in a straight line

**3.4**
**parallel length**
$L_c$
length of the parallel reduced section of the test piece

**3.5**
**extensometer gauge length**
$L_e$
distance between the measuring points of the extensometer

NOTE    In some cases, $L_e = L_o$ and may also be used for the calculation of elongation.

**3.6**
**original cross-sectional area**
$S_o$
cross-sectional area of the parallel length as determined at ambient temperature prior to testing

**3.7**

**minimum cross-sectional area after rupture**

$S_u$

minimum cross-sectional area of the parallel length as determined at ambient temperature after rupture, with the pieces carefully fitted back together with their axes in a straight line

**3.8**

**initial stress**

$\sigma_o$

applied force divided by the original cross-sectional area ($S_o$) of the test piece

**3.9**

**elongation**

$\Delta L_r$

increase of the reference length ($L_r$)

NOTE    See 6.2.

**3.10**

**percentage elongation**

$A$

elongation expressed as a percentage of the original reference length ($L_{ro}$)

NOTE 1    See Figure 1.

NOTE 2    In the terms for elongation in 3.10 to 3.16, the symbol "$\varepsilon$" may replace "$A$".

However, when "$\varepsilon$" is used, the following conventions should apply:

$\varepsilon\,\%$        is the percentage strain or elongation;

$\varepsilon$         is the absolute strain.

**3.11**

**percentage initial plastic elongation**

$A_i$

non-proportional increase of the original reference length ($L_{ro}$) due to the application of the test force

**3.12**

**percentage creep elongation**

$A_f$

increase in reference length at time $t$ ($\Delta L_{rt}$) at a specified temperature expressed as a percentage of the original reference length ($L_{ro}$):

$$A_f = \frac{\Delta L_{rt}}{L_{ro}} \times 100$$

(1)

NOTE 1    $A_f$ may have the specified temperature ($T$) in degrees Celsius (°C) as superscript and the initial stress ($\sigma_o$) in megapascals [2] and time $t$ (in hours) as subscript.

NOTE 2    By convention, the beginning of creep elongation measurement is the time at which the initial stress ($\sigma_o$) is applied to the test piece (see Figure 1).

NOTE 3    Suffix f originates from "fluage", "creep" in French.

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2)    1 MPa = 1 N/mm².

**3.13**
**percentage plastic elongation**

$A_p$

non-proportional increase of the original reference length ($L_{ro}$) at time $t$:

$$A_p = A_i + A_f \tag{2}$$

**3.14**
**percentage anelastic elongation**

$A_k$

non-proportional decrease of the original reference length ($L_{ro}$) at time $t$ due to unloading

**3.15**
**percentage permanent elongation**

$A_{per}$

total increase of the original reference length ($L_{ro}$) at time $t$ determined after unloading:

$$A_{per} = A_p - A_k \tag{3}$$

**3.16**
**percentage elongation after creep rupture**

$A_u$

permanent increase of the original reference length ($L_{ro}$) after rupture ($L_{ru} - L_{ro}$) expressed as a percentage of the original reference length ($L_{ro}$):

$$A_u = \frac{L_{ru} - L_{ro}}{L_{ro}} \times 100 \tag{4}$$

NOTE     $A_u$ may have the specified temperature ($T$) in degrees Celsius as superscript and the initial stress ($\sigma_o$) in megapascals as subscript.

**3.17**
**percentage reduction of area after creep rupture**

$Z_u$

maximum change in cross-sectional area measured after rupture ($S_o - S_u$) expressed as a percentage of the original cross-sectional area ($S_o$):

$$Z_u = \frac{S_o - S_u}{S_o} \times 100 \tag{5}$$

NOTE     $Z_u$ may have the specified temperature ($T$) in degrees Celsius as superscript and the initial stress ($\sigma_o$) in megapascals as subscript.

**3.18**
**creep elongation time**

$t_{fx}$

time required for a strained test piece to obtain a specified percentage creep elongation ($x$) at the specified temperature ($T$) and the initial stress ($\sigma_o$)

EXAMPLE   $t_{f0,2}$.

**3.19**
**plastic elongation time**

$t_{px}$

time required to obtain a specified percentage plastic elongation ($x$) at the specified temperature ($T$) and the initial stress ($\sigma_o$)

**3.20**
**creep rupture time**
$t_u$
time to rupture for a test piece maintained at the specified temperature ($T$) and the initial stress ($\sigma_o$)

NOTE    The symbol $t_u$ may have as superscript the specified temperature ($T$) in degrees Celsius and as subscript the initial stress ($\sigma_o$) in megapascals.

**3.21**
**single test piece machine**
testing machine that permits straining of a single test piece

**3.22**
**multiple test piece machine**
testing machine that permits straining of more than one test piece simultaneously at the same temperature

# 4    Symbols and designations

The symbols and corresponding designations are given in Table 1.

**Table 1 — Symbols and designations**

| Symbol [a] | Unit | |
|---|---|---|
| $D$ | mm | Diameter of the cross-section of the parallel length of a cylindrical test piece |
| $D_n$ | mm | Diameter of gauge length containing a notch |
| $d$ | mm | Diameter of gauge length without a notch in a combined notched/un-notched test piece (see Figure C.1) |
| $d_n$ | mm | Diameter across root of circumferential notch<br>For a combined notched/un-notched test piece $d = d_n$ |
| $b$ | mm | Width of the cross-section of the parallel length of a test piece of square or rectangular cross-section |
| $L_r$ | mm | Reference length |
| $a$ | mm | Thickness of a test piece of square or rectangular cross-section [see Figure 2 b)] |
| $L_{ro}$ | mm | Original reference length |
| $L_{ru}$ | mm | Final reference length |
| $\Delta L_r$ | mm | Elongation |
| $\Delta L_{rt}$ | mm | Increase in reference length at time $t$ |
| $L_o$ | mm | Original gauge length |
| $L_n$ | mm | Parallel gauge length containing a notch |
| $L_u$ | mm | Final gauge length after rupture |
| $L_c$ | mm | Parallel length |
| $L_e$ | mm | Extensometer gauge length |
| $R$ | mm | Transition radius |
| $r_n$ | mm | Notch root radius |
| $S_o$ | mm² | Original cross-sectional area of the parallel length |
| $S_u$ | mm² | Minimum cross-sectional area after rupture |

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Table 1 (*continued*)

| Symbol [a] | Unit | Designation |
|---|---|---|
| $\sigma_o$ | MPa | Initial stress |
| $A_e$ [b] | % | Percentage elastic elongation |
| $A_i$ [b] | % | Percentage initial plastic elongation |
| $A_k$ [b] | % | Percentage anelastic elongation |
| $A_p$ [b] | % | Percentage plastic elongation |
| $A_{per}$ [b] | % | Percentage permanent elongation |
| $A_f$ [b] | % | Percentage creep elongation: $$A_f = \frac{\Delta L_{rt}}{L_{ro}} \times 100$$ NOTE     As an example, the symbol may be completed as follows: $A_{f50/5\,000}^{375}$ : percentage creep elongation with an initial stress of 50 MPa after 5 000 h at the specified temperature of 375 °C. |
| $A_u$ [b] | % | Percentage elongation after creep rupture: $$A_u = \frac{L_{ru} - L_{ro}}{L_{ro}} \times 100$$ NOTE     As an example, the symbol may be completed as follows: $A_{u50}^{375}$ : percentage elongation after creep rupture with an initial stress of 50 MPa at the specified temperature of 375 °C. |
| $Z_u$ | % | Percentage reduction of area after creep rupture: $$Z_u = \frac{S_o - S_u}{S_o} \times 100$$ NOTE     As an example, the symbol may be completed as follows: $Z_{u50}^{375}$ : percentage reduction of area after creep rupture with an initial stress of 50 MPa at the specified temperature of 375 °C. |
| $t_{fx}$ | h | Creep elongation time |
| $t_{px}$ | h | Plastic elongation time |
| $t_u$ | h | Creep rupture time NOTE     As an example, the symbol may be completed as follows: $t_{u50}^{375}$ : creep rupture time with an initial stress of 50 MPa at the specified temperature of 375 °C. |
| $t_{un}$ | h | Creep rupture time of a notched test piece |
| $T$ | °C | Specified temperature |
| $T_i$ | °C | Indicated temperature |
| $x$ | % | Specified percentage creep or plastic elongation |
| $n$ | | Creep exponent |

[a]     The main subscripts (r, o and u) of the symbols are used as follows:

r    corresponds to reference;

o    corresponds to original;

u    corresponds to ultimate (after rupture).

[b]     See Note 2 in 3.10.

## 5   Principle

The test consists of heating a test piece to the specified temperature and of straining the test piece by means of a constant tensile force or constant tensile stress (see note) applied along its longitudinal axis for a period of time to obtain any of the following:

— a specified creep elongation (uninterrupted test);

— values of permanent elongation at suitable intervals throughout the test (interrupted test);

— the creep rupture time (uninterrupted and interrupted test).

NOTE       "Constant stress" means that the ratio of the force to the instantaneous cross-section remains constant throughout the test. The results obtained with constant stress are generally different from those obtained with constant force.

## 6   Apparatus

### 6.1   Testing machine

The testing machine shall apply a force along the axis of the test piece while keeping inadvertent bending or torsion of the test piece to a minimum. Prior to test the machine should be visually examined to ensure that loading bars, grips, universal joints and associated equipment are in a good state of repair.

The force should be applied to the test piece without shock.

The machine should be isolated from external vibration and shock. The machine should be equipped with a device which minimizes shock when the test piece ruptures.

NOTE       At present, there appears to be insufficient quantitative data in the literature demonstrating the influence of bending upon creep and stress rupture life. It is requested that any organization with such information forwards it to ISO/TC164 for consideration at the next revision of this International Standard.

The machine shall be verified and shall meet the requirements of at least class 1 in ISO 7500-2.

### 6.2   Elongation measuring device

In uninterrupted tests, the elongation shall be measured using an extensometer, which meets the performance requirements of class 1 or better of ISO 9513 or by other means which ensure the same accuracy without interruption of the test. The extensometer can either be directly attached to the test piece, or can be non-contacting (e.g. a non-contacting optical or laser extensometer).

It is recommended that the extensometer is calibrated over an appropriate range based upon the expected creep strain.

The extensometer shall be calibrated at intervals not exceeding 3 years, unless the test duration is longer than 3 years. If the predicted test exceeds the date of the expiry of the calibration certificate then the extensometer shall be recalibrated prior to commencement of the creep test.

The extensometer gauge length shall not be less than 10 mm.

The extensometer shall be able to measure the elongation either on one side or on the opposite sides of the test piece; the latter is the preferred option.

The type of extensometer used (e.g. single-sided, double-sided, axial, diametral) should be reported. When the elongation is measured on the opposite sides, the average elongation should be reported.

NOTE 1    For uninterrupted creep tests, i.e. with an extensometer attached directly to the parallel section of a test piece, the percentage creep elongation is measured over $L_e$.

When the elongation is measured with an extensometer attached to the grip ends of the test piece, the ends shall be of such shape and size that it can be assumed that the observed elongation has occurred completely within the reference length of the test piece. Percentage creep elongation is measured over $L_r$.

The extensometer gauge length should normally be as near as possible to the reference length. In the case of accurate creep measurements, a gauge length as long as possible should be used to improve the accuracy of measurements.

NOTE 2    If only the percentage elongation after creep rupture or the percentage creep elongation for a specified test duration is determined, the use of an extensometer is not necessary.

In interrupted tests, periodically unload the test piece and cool it to ambient temperature and measure the permanent elongation on the gauge length with an appropriate device. The precision of this device shall be 0,01 $\Delta L_r$ or 0,01 mm, whichever is the greater. After this measurement the test piece may be first reheated and then reloaded.

NOTE 3    For low creep strain measurements, e.g. $\leqslant 1\%$ strain, on test pieces with short gauge lengths, careful consideration needs to be given to ensure that the measuring device used has sufficient resolution.

NOTE 4    Information on the long-term stability of transducers used for creep testing and accreditation issues are given in References [35] and [36] in the Bibliography.

Care should be taken to avoid spurious negative creep when using nickel base alloy extensometers. See the Code of Practice by Loveday and Gibbons (2007) [38].

## 6.3    Heating device

### 6.3.1    Permissible temperature deviations

The heating device shall heat the test piece to the specified temperature $(T)$. The permitted deviations between the indicated temperature, $(T_i)$ and the specified temperature, $(T)$, and the permitted maximum temperature variation along the test piece shall be as given in Table 2.

**Table 2 — Permitted deviations between $T_i$ and $T$**
**and maximum permissible temperature variation along the test piece**

| Specified temperature, $T$<br>°C | Permitted deviation between $T_i$ and $T$<br>°C | Maximum permissible temperature variation along the test piece<br>°C |
|:---:|:---:|:---:|
| $T \leqslant 600$ | ± 3 | 3 |
| $600 < T \leqslant 800$ | ± 4 | 4 |
| $800 < T \leqslant 1\,000$ | ± 5 | 5 |
| $1\,000 < T \leqslant 1\,100$ | ± 6 | 6 |

For specified temperatures greater than 1 100 °C, the permitted values shall be defined by agreement between the parties concerned.

The indicated temperatures $(T_i)$ are the temperatures measured at the surface of the parallel length of the test piece, errors from all sources being taken into account and any systematic errors having been corrected.

NOTE      Instead of measuring the temperature at the surface of the test piece, it is permitted to carry out indirect measurement of the temperature of each heating zone of the furnace provided that it is demonstrated that the tolerance defined above is fulfilled.

If an extensometer is used, the parts of this instrument outside the furnace shall be designed and protected in such a way that the temperature variations in the air around the furnace do not significantly affect the measurements of the variations in length.

Variations in temperature of the air surrounding the test machine should not exceed $\pm 3\ °C$.

In the interrupted test, the variation of the room temperature during all measurements of the gauge length should not exceed $\pm 2\ °C$. If this range is exceeded, corrections for ambient temperature variations shall be applied.

## 6.3.2   Temperature measurement

### 6.3.2.1   General

The temperature indicator shall have a resolution of at least 0,5 °C and the temperature measuring equipment shall have an accuracy equal to or better than $\pm 1\ °C$.

### 6.3.2.2   Single test piece machines

In single test piece machines, for test pieces with a parallel length less than or equal to 50 mm, at least two thermocouples should be used. For test pieces with a parallel length greater than 50 mm, at least three thermocouples should be used. In all cases, a thermocouple should be placed at each end of the parallel length and, if a third thermocouple is used, it should be placed in the middle region of the parallel length.

The number of thermocouples may be reduced to one if it can be demonstrated that the conditions of the furnace and the test piece are such that the variation of temperature of the test piece does not exceed the values specified in 6.3.1.

### 6.3.2.3   Multiple test piece machines

In multiple test piece machines, it is recommended that at least one thermocouple be used for each test piece. If only one thermocouple is used it shall be positioned at the middle of the parallel length. Three thermocouples may only be used if located at appropriate positions within the furnace, and if there is supporting data to demonstrate that for all test pieces the temperature conforms to the requirements of 6.3.1.

In the case of indirect temperature measurement, regular control measurements are required to determine differences between the thermocouple(s) of each heating zone and a significant number of test pieces within a given zone. The non-systematic components of the temperature differences shall not exceed $\pm 2\ °C$ up to 800 °C and $\pm 3\ °C$ above 800 °C.

### 6.3.2.4   Notched test pieces

Temperature measurement of notched test pieces shall be in accordance with either 6.3.2.2 or 6.3.2.3. It is recommended that one thermocouple is placed close to the notch.

### 6.3.2.5   Thermocouples

The thermocouple junctions shall make good thermal contact with the surface of the test piece and shall be screened from direct radiation from the heating source. The remaining portions of the wires within the furnace shall be thermally shielded and electrically insulated.

NOTE      This clause is not applicable in the case of indirect temperature measurement.

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### 6.3.3 Calibration of the thermocouples and temperature measuring system

NOTE    Information concerning different types of thermocouples is given in Annex A.

#### 6.3.3.1 Calibration of the thermocouples

Rare metal thermocouples in use for short duration tests (typically 500 h or less) should be calibrated at least every 12 months. Thermocouples in use for test durations greater than 12 months should be calibrated as follows:

— 4 years for $T \leqslant 600$ °C;

— 2 years for 600 °C $< T \leqslant 800$ °C;

— 1 year for $T > 800$ °C.

If a test duration exceeds the above calibration period the thermocouple shall be calibrated upon completion of the test. If a thermocouple is rewelded, the thermocouple shall be recalibrated before use.

It shall be demonstrated that the error of the thermocouple used has been established either at the test temperature or is typical for a range containing the test temperature.

If it is demonstrated that the drift of the thermocouple does not affect the permissible temperature deviations specified in 6.3.1, the period between two calibrations can be longer.

Changes in the output of a thermocouple can be due not only to chemical changes from contamination leading to drift, but also as a consequence of handling physical damage. Information on such changes should be recorded and should be available on request.

NOTE 1    Thermocouple drift is dependent on the type of thermocouple used and the exposure duration at temperature.

If the drift affects permissible temperature deviations, either more frequent calibrations should be carried out or a correction may be made to the temperature indicated by the thermocouple.

NOTE 2    Information concerning methods of calibration of thermocouples is given in Annex B.

#### 6.3.3.2 Calibration of the temperature measuring equipment

The calibration of the temperature measuring equipment (including the cable, the connection, the cold junction, the indicator or the recorder, the data line, etc.) shall be carried out by a method traceable to the international unit (SI) of temperature.

If practicable, this calibration should be carried out annually over the range of temperatures measured by the equipment and the readings shall be given in the calibration report.

## 7   Test pieces

### 7.1   Shape and dimensions

In general, the test piece is a machined proportional cylindrical test piece ($L_{ro} = k\sqrt{S_o}$) with a circular cross-section (see Figure 2). The value $k$ should be equal to or greater than 5,65 and the value used shall be recorded in the test report, i.e. $L_{ro} \geqslant 5D$.

In special cases, the cross-section of the test piece may be square or rectangular or of some other shape. The provisions concerning the cylindrical test pieces do not apply to these specific test pieces.

In general, $L_{ro}$ should not exceed $L_c$ by more than 10 % for circular test pieces, or by more than 15 % for square or rectangular test pieces.

The parallel length shall be joined by transition curves to the gripped ends, which may be of any shape to suit the grips of the testing machine. The transition radius ($R$) should be between $0,25D$ and $1D$ for the cylindrical test pieces, or $0,25b$ and $1b$ in the case of rectangular or square test pieces.

Unless the sample size does not permit it, the original cross-sectional area ($S_o$) shall be greater than or equal to 7 mm².

NOTE    In some cases, especially for brittle materials, the transition radius can be greater than $1D$.

When a test piece having extensometer attachment ridges (collars) in the parallel length is used, the transition radius of the collars may be less than $0,25d$; this should be selected to minimize stress concentrations and there should be no evidence of undercut when inspected. For test pieces with collars, the diameter between the collar and the grip end may be up to 10 % larger than the diameter of the original gauge length; this should ensure that fracture will occur within the gauge length.

The grip ends of test pieces shall have the same axis as the parallel length with a coaxiality tolerance of

— $0,005D$ or 0,03 mm, whichever is greater, for cylindrical test pieces, and

— $0,005b$ or 0,03 mm, whichever is greater, for rectangular or square test pieces.

When oxidation is a significant factor, test pieces with a larger original cross-sectional area ($S_o$) should be used.

The original reference length shall be determined to a measurement uncertainty of ± 1 %. The final reference length should be determined to a measurement uncertainty of ± 1 %.

When a notched test piece is used, the geometry and the position of this notch should be defined by agreement.

## 7.2   Preparation

The test piece shall be machined in such a way as to minimize any residual deformation or surface defects.

The shape tolerances shall conform to Table 3 for test pieces with circular cross-sections and to Table 4 for test pieces with square or rectangular cross-sections.

**Table 3 — Shape tolerances of test pieces with circular cross-sections**

Dimensions in millimetres

| Nominal dimension $D$ | Shape tolerances [a] |
|---|---|
| $3 < D \leqslant 6$ | 0,02 |
| $6 < D \leqslant 10$ | 0,03 |
| $10 < D \leqslant 18$ | 0,04 |
| $18 < D \leqslant 30$ | 0,05 |
| [a]    Maximum deviation between the measurements of a transverse dimension determined along the entire parallel length of the test piece (see ISO 286-2). | |

**Table 4 — Shape tolerances of test pieces with square or rectangular cross-sections**

Dimensions in millimetres

| Nominal dimension $b$ | Shape tolerances [a] |
|---|---|
| $3 < b \leqslant 6$ | 0,02 |
| $6 < b \leqslant 10$ | 0,03 |
| $10 < b \leqslant 18$ | 0,04 |
| $18 < b \leqslant 30$ | 0,05 |
| [a]   Maximum deviation between the measurements of a transverse dimension determined along the entire parallel length of the test piece (see ISO 286-2). | |

It is recommended that the minimum original cross-sectional area should occur within the middle two thirds of the parallel length or of the reference length, whichever is smaller.

When the test piece has a notch (see Annex C), its profile shall be checked to ensure that it conforms with the tolerances specified in the relevant product standard.

## 7.3   Determination of the original cross-sectional area

The original cross-sectional area $(S_o)$ shall be calculated from measurement of appropriate dimensions within the parallel length. Each appropriate dimension shall be measured to a measurement uncertainty of $\pm\,0,1$ % or 0,01 mm, whichever is greater.

The size of the test piece shall be determined at three positions along the gauge length and the minimum calculated value of the cross-sectional area shall be used for determining the applied load corresponding to the specified stress.

## 7.4   Marking of the original gauge length, $L_o$

Each end of the original gauge length shall be marked by means of fine marks or scribed lines, or other means, but not by notches which could result in premature fracture.

Where marked, the original gauge length shall be marked to an accuracy of $\pm\,1$ %.

NOTE      In some cases, it may be helpful to draw, on the surface of the test piece, a line parallel to the longitudinal axis, along which the gauge length is drawn. Marking of $L_o$ is not necessary when a specimen with small collars is used [see Figure 2 c)].

## 7.5   Determination of the reference length, $L_r$

Where extensometry is attached to either ridges on the parallel length or the shoulders of the test piece, the reference length shall be calculated using the following equation:

$$L_r = L_c + 2\sum_i \left[ \left( D / d_i \right)^{2n} l_i \right] \tag{6}$$

See Figure 2 e), where

$n$   is the stress exponent at the test temperature for the material under investigation. If this is not known, use $n = 5$; and

$l_i$    is the length increment in the transition region. Experience has shown a value of 0,1 mm to be suitable for these calculations.

This calculation shall be performed for each test piece design; providing the test piece dimensions remain within the limits defined in 7.1 and 7.2 a recalculation for each test piece produced to that design is not required.

# 8   Test procedure

## 8.1   Heating of the test piece

The test piece shall be heated to the specified temperature ($T$). The test piece, gripping device and the extensometer shall be at thermal equilibrium.

This condition shall be maintained for at least one hour before application of the force to the test piece, unless the product standard states otherwise. In the uninterrupted test, the maximum time that the test piece is held at the test temperature before applying the force shall not exceed 24 h. In the interrupted test, this time should not exceed 3 h; the time under test temperature without force after unloading should not exceed 1 h.

During the heating period, the temperature of the test piece should not, at any time, exceed the specified temperature ($T$) with its tolerances. If these tolerances are exceeded, it shall be reported.

For creep tests with extensometers, a small preload (less than 10 % of the test force) may be applied to the test piece in order to keep the loading train in alignment whilst heating up the test piece (i.e. before $t = 0$).

## 8.2   Application of the test force

The test force shall be applied along the test axis in such a manner to minimize bending and torsion of the test piece.

The applied force shall be known to an accuracy of at least $\pm 1$ %. The application of the test force shall be made without shock and should be as rapid as possible.

Special care should be taken during the loading of soft and face centred cubic (FCC) materials since they may exhibit creep at very low loads or at room temperature.

The beginning of the creep test and measurement of creep elongation is the time ($t = 0$) when the full load of the initial stress is applied to the test piece (see Figure 1).

## 8.3   Test interruptions

### 8.3.1   General

The number of periodic interruptions should be sufficient to obtain the elongation data.

### 8.3.2   Multiple test piece machine with several test pieces in line

After a test piece has ruptured, the string of test pieces shall be removed from the testing machine to allow replacement. Resume testing in accordance with 8.1 and 8.2.

### 8.3.3   Accidental interruption of the test

For any accidental interruption of the test due to, for example, interruption of heating or current, the conditions of resumption of the test after each interruption shall be recorded in the test report. Ensure that overloading of the test piece due to contraction of the force assembly is prevented. It is recommended that the initial applied force is maintained during these interruptions.

## 8.4 Recording of temperature and elongation

### 8.4.1 Temperature

Throughout the test, it is important that sufficient recordings of the temperature of the test piece are made to demonstrate that the temperature conditions comply with the requirements of 6.3.1.

### 8.4.2 Elongation

Either a continuous record or a sufficient number of recordings of the elongation shall be made throughout the test so that the creep-time curve can be traced (see Figure 3).

When only a determination of a creep elongation for a specified test duration is made, the drawing of the creep-time diagram is not necessary. Only the initial and final measurements are required.

In the interrupted test, the number of periodic interruptions for elongation measurement shall be chosen in order to make it possible to interpolate the creep-time curve with sufficient accuracy to determine times to percentage permanent elongation.

In the uninterrupted test, the percentage initial plastic elongation, $A_i$, shall be determined.

NOTE    If the sum of elastic and initial plastic elongation is measured, the elastic elongation to be subtracted can be determined from a stepwise measurement procedure during loading, or from a partial unloading procedure during the test, or it can be taken over from a tensile test at elevated temperature in accordance with ISO 783 with a similar loading rate as in the creep test.

To determine the percentage initial plastic elongation, $A_i$, in the case of the interrupted creep test, a tensile test at elevated temperature in accordance with ISO 783 shall additionally be performed at each creep test temperature and with a similar loading rate as in the creep test.

EXAMPLE    An example of a sequence of time intervals for interruption strain measurements for long-term testing is: 100 h, 250 h, 1 000 h, 2 500 h, 5 000 h, every 5 000 h until 40 000 h then every 10 000 h thereafter.

Tests of 3 000 h duration or less should have an additional interruption at 50 h; for tests of 1 000 h or less a further interruption at 25 h should be included in the test plan.

### 8.4.3 Elongation time diagram

On the basis of records of time and elongation, an elongation-time diagram can be drawn (see Figure 3).

## 9 Determination of results

The test results are determined from the preceding recordings using the definitions given in Clause 3.

## 10 Test validity

Unless the results meet the requirements of the product standard or the customer specification, the rupture elongation shall be considered invalid if the test piece ruptures outside the parallel length ($L_c$) or outside the extensometer gauge length ($L_e$).

## 11 Accuracy of the results

### 11.1 Expression of the results

For the expression of the results, the values shall be expressed taking into account the following requirements concerning the rounding rules:

— specified temperature ($T$):    to 1 °C;

— diameter ($D$):    to 0,01 mm;

— ratio ($L_{ro}/D$):    to one decimal place;

— original reference length ($L_{ro}$):    to 0,1 mm;

— initial stress ($\sigma_o$):    3 significant figures;

— time ($t_{fx}$, $t_{px}$):    3 significant figures;

— time ($t_u$, $t_{un}$):    1 %, or to the nearest hour, whichever is smaller;

— percentage elongation ($A_e$, $A_i$, $A_f$, $A_p$, $A_{per}$, $A_k$):    3 significant figures;

— percentage elongation after creep rupture ($A_u$):    2 significant figures;

— percentage reduction of area after creep rupture ($Z_u$):    2 significant figures.

### 11.2 Final uncertainty

Due to the fact that the uncertainty of the results depends on the nature of the tested material and the testing conditions, it is not possible to give precise values for the uncertainty.

Examples of estimated uncertainty for some materials are given in Annex D.

## 12 Test report

**12.1**  Information on materials not covered by a product specification shall be reported in accordance with 12.2, or both 12.2 and 12.3. For a representation of results and graphical extrapolation, see Annex E.

**12.2**  Information to be reported in the test report shall include, when applicable:

— reference to this International Standard;

— type of test (uninterrupted or interrupted);

— material and test piece identification;

— type and dimensions of the test piece (value of the proportionality coefficient $k$ included), including the reference length used;

— specified temperature and indicated temperature, if it is outside the permitted limits;

— initial applied stress;

— constant applied force or constant applied stress;

— test results;

— position of the rupture (when outside of central two thirds of the parallel length);

— percentage initial plastic elongation;

— conditions of accidental interruptions and resumptions of the test;

— any occurrence which can affect the results, for example, deviations from the specified tolerances.

**12.3** Information to be available on request (made at the time of order) may include, when applicable:

— machine type (simple machine, multiple machine with test pieces in line, etc.);

— force application time;

— elongation-time diagram with sufficient recordings to accurately construct the diagram;

— percentage elastic elongation due to the application of the force (see 8.4.2);

— percentage elastic and anelastic elongation due to unloading and the unloading time (see 8.4.2);

— information concerning the recorded values of any indicated temperature excursions outside the permitted temperature limits defined in 6.3.1;

— type of extensometer;

— value of the drift of the thermocouples over the test period;

— see also E.6 for recommended additional information regarding the sample material.

**12.4** The test conditions and limits defined in this International Standard shall not be adjusted to take account of uncertainties of measurement, unless specifically instructed otherwise by the customer (see Annex D).

**12.5** The estimated measurement uncertainties shall not be combined with test results to assess compliance with product specifications, unless specifically instructed otherwise by the customer (see Annex D).
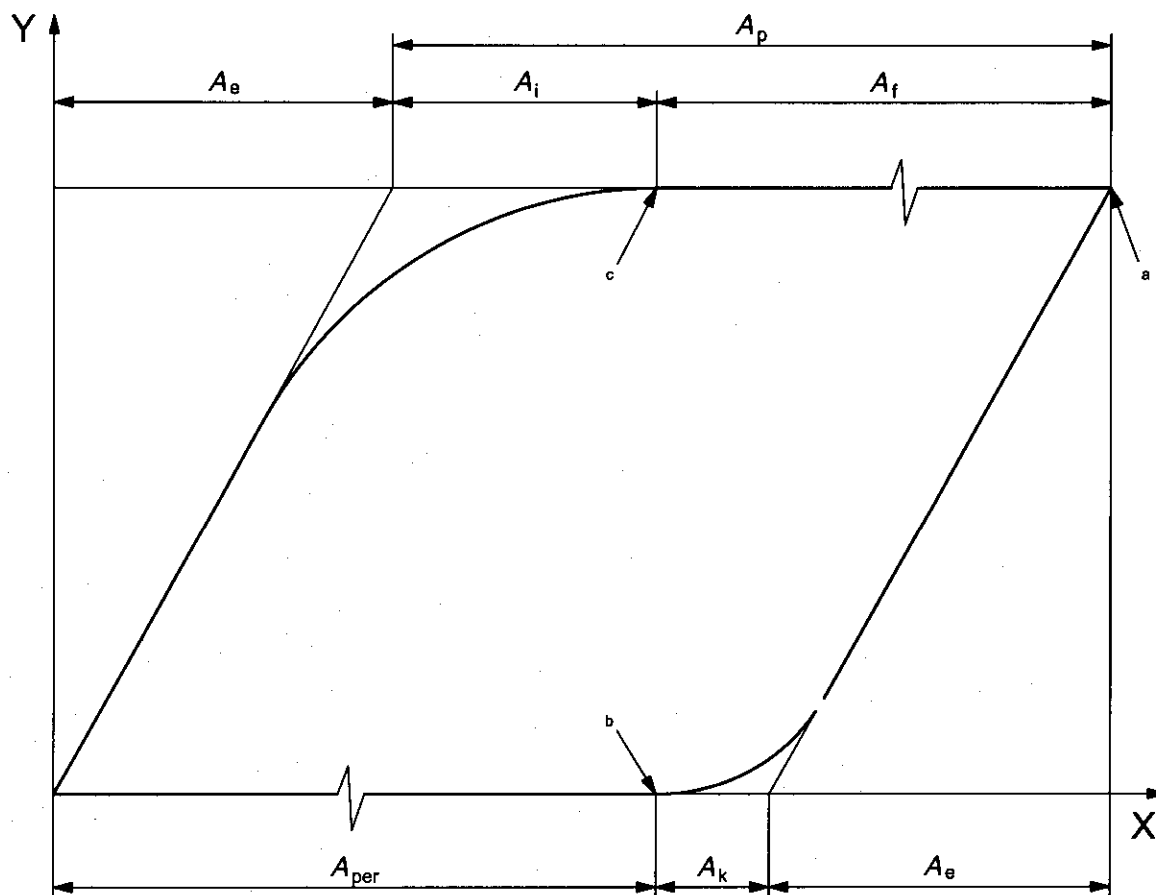
**Key**

| | |
|---|---|
| X | elongation |
| Y | stress |
| $A_e$ | elastic elongation |
| $A_f$ | creep elongation |
| $A_i$ | initial plastic elongation |
| $A_k$ | anelastic elongation |
| $A_p$ | total plastic elongation |
| $A_{per}$ | permanent elongation |
| [a] | Start of unloading. |
| [b] | End of unloading. |
| [c] | End of loading [$t = 0$ (zero moment)]. |

**Figure 1 — Schematic stress-elongation diagram**
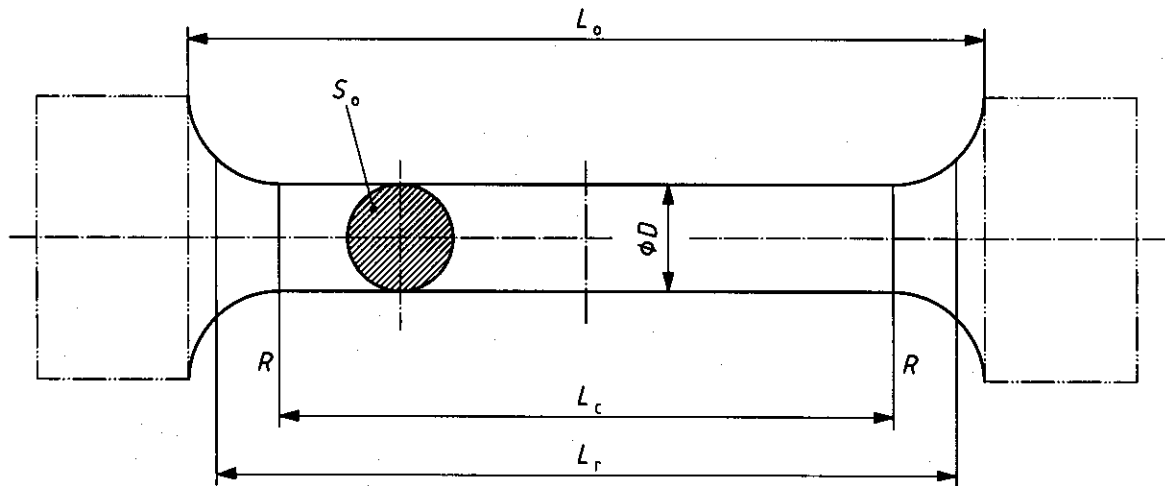
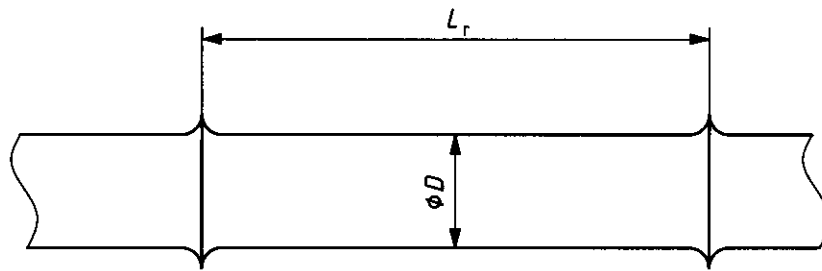NOTE $L_r$ should be determined in accordance with Equation (6).

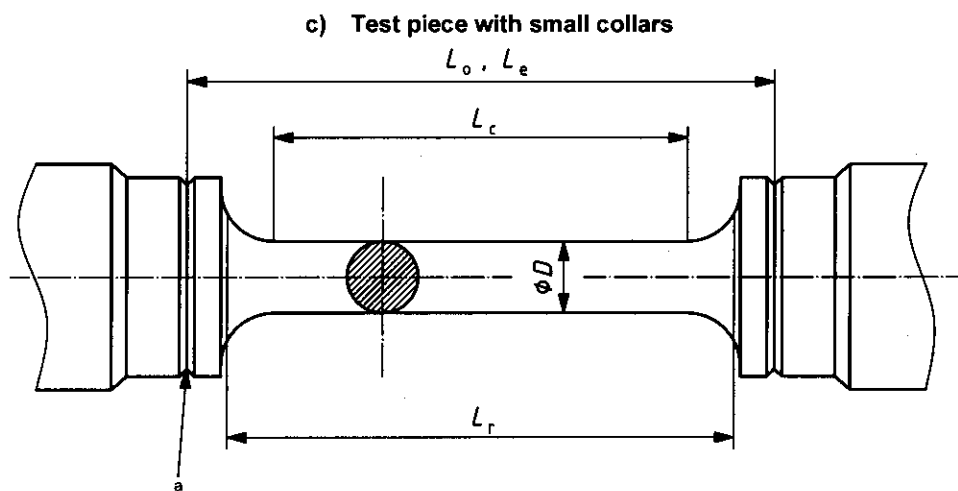**a)  Test piece with shoulders and gauge length outside parallel length**
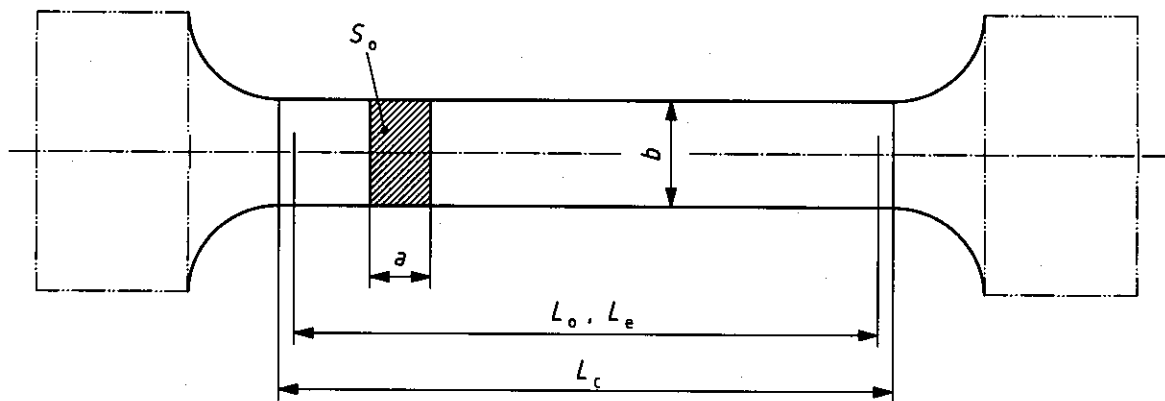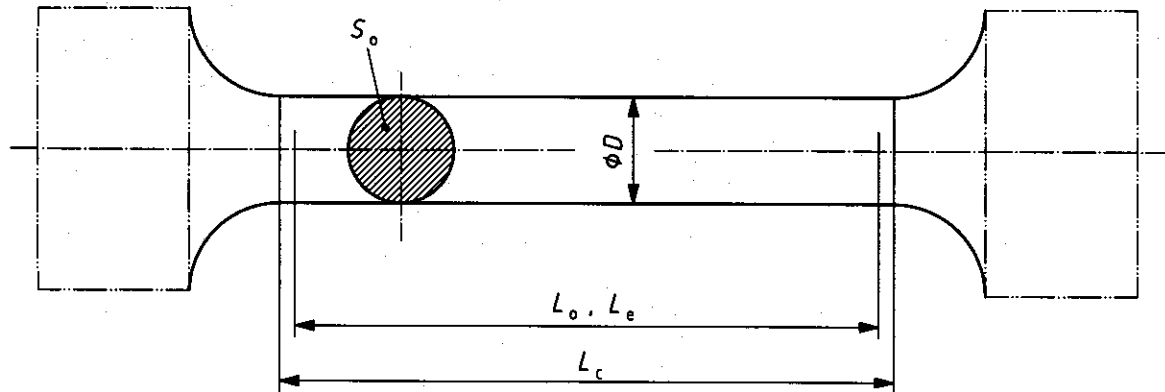


**b)  Test piece with shoulders and gauge length inside parallel length**

**Figure 2** (*continued*)
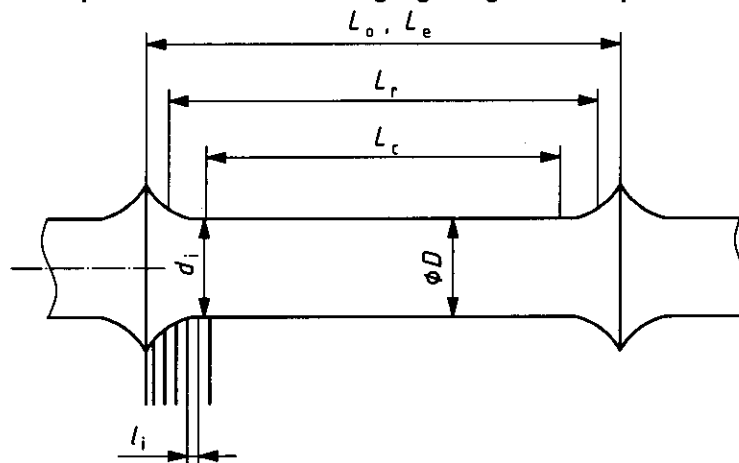
NOTE    Generally $L_r$ is equal to $L_o$ or $L_e$.

### c)  Test piece with small collars



**Key**

a    V notch (angle between 55° and 90°, depth 0,15 mm)

NOTE    $L_r$ should be determined in accordance with Equation (6).

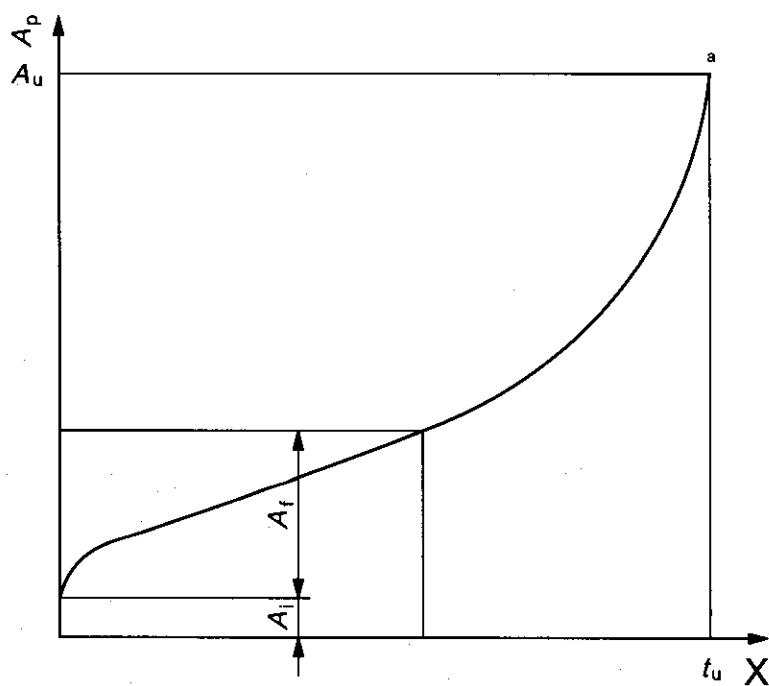### d)  Test piece with shoulders and gauge length outside parallel length



### e)  Test piece with collars

**Figure 2 — Examples of test pieces** [3]

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3)  The shape of the grip ends is only given for information.

**Key**

X    time from the end of loading

a    Rupture.

**Figure 3 — Elongation-time diagram**

# Annex A
## (informative)

# Information concerning different types of thermocouples

Information concerning different types of thermocouples is given in IEC 60584-1 [1] and IEC 6084-2 [2].

The use of rare metal thermocouples, preferentially of type S or R, is recommended for temperatures equal to or greater than 400 °C.

Base metal thermocouples of type K should only be used either for temperatures lower than 400 °C or for times less than 1 000 h at higher temperatures, and should not be re-used.

Base metal thermocouples of type N may be used either for temperatures lower than 600 °C or for times less than 3 000 h at higher temperatures, and should not be re-used.

The thermocouple drift should not exceed the following values within the calibration period:

— $\pm 1$ °C for $T \leqslant 600$ °C;

— $\pm 1,5$ °C for $600$ °C $< T \leqslant 800$ °C;

— $\pm 2$ °C for $800$ °C $< T \leqslant 1\ 100$ °C.

For rare metal thermocouples, these requirements are in general fulfilled for the following calibration periods:

— 4 years for $T \leqslant 600$ °C;

— 2 years for $600$ °C $< T \leqslant 800$ °C;

— 1 year for $800$ °C $< T \leqslant 1\ 100$ °C.

# Annex B
## (informative)

## Information concerning methods of calibration of thermocouples

For the thermocouple calibration, two strategies can be recommended. The objective of both is to ensure that the electromagnetic field (EMF) indicated by the thermocouple at the calibration temperature (corrected, where necessary, for all systematic errors) equates as closely as possible to the EMF defined by the appropriate IEC 60584-1 [1] reference table for that temperature. Both strategies employ the use of reference thermocouples, which are directly traceable to a national standard. A pre-requisite is that the calibration tolerance of the new thermocouple is in accordance with IEC 60584-2 [2], class 1 or an equivalent standard. The calibration of the temperature measuring equipment can be carried out separately or during the thermocouple calibration.

Strategy 1 is based on *in situ* calibration of the thermocouple, i.e. thermocouple calibration either in the actual furnace or in a calibration furnace with the same depth of immersion and temperature gradient along the thermocouple wires. The error determined during *in situ* calibration is used to correct the specified temperature of the thermocouple. If the error exceeds the limit associated with the uncertainty relating to the immersion depth, the thermocouple is scrapped. Reference thermocouple drift due to variable immersion depth during active and passive service should be surveyed and minimized.

Strategy 2 involves calibration of the thermocouple in a calibration furnace in which the depth of immersion is similar to that in the testing furnace. If, on calibration, the laboratory's tolerance, which needs to include the effect due to depth of immersion, is exceeded, the thermocouple is cut back and re-welded at the hot junction and/or annealed and calibration repeated. If after repeated calibration, the laboratory's calibration tolerance remains exceeded, the thermocouple is scrapped.

# Annex C
## (normative)

# Creep testing using test pieces with V or blunt circumferential notches

## C.1 General

Circumferentially notched test pieces may be employed in tensile creep testing to provide either

a) the material response to a feature that introduces a significant stress concentration, e.g. a sharp change in section of a component such as the root of a thread, or

b) the response of the material under a multi-axial stress state.

The former (a) may be evaluated using a V notch geometry as discussed in C.2, whilst the latter may be achieved using blunt, or semi-circular circumferential notches as discussed in C.3.

## C.2 V-notched test pieces

The use of circumferential V-notched test pieces has long been used to determine a material's response to features such a threads in components, both in tensile and creep testing. Frequently, a combined test piece geometry was employed having a parallel shank region with the same cross-sectional area as that across the throat of a notch machined into a larger diameter portion of the same test piece (see Figure C.1). Such test pieces were primarily used to determine whether the material 'notch strengthened', i.e. fractured in the plain shank region first, or 'notch weakened', i.e. fractured across the notch. Clearly, the magnitude of the notch strengthening or weakening effect could not be quantified from the use of the combined test piece geometry and if such information is required it is necessary to test separately plain and notched test pieces under the same net section stress.

**Table C.1 — Examples dimensions of notched specimens with circular cross-sections and with an elastic stress concentration factor $K_t = 4,5 \pm 0,5$ [3]**

Dimensions in millimetres

| Root diameter, $d_n$ Tolerances $\pm 0,02$ | Shaft diameter, $D_n$ Tolerances $\pm 0,1$ | Notch radius, $r_n$ | Tolerances on $r_n$ |
|---|---|---|---|
| $3 < d_n \leqslant 6$ | $4 < D_n \leqslant 8$ | $0,07 < r_n \leqslant 0,14$ | $\pm 0,02$ |
| $6 < d_n \leqslant 10$ | $8 < D_n \leqslant 13,3$ | $0,14 < r_n \leqslant 0,24$ | $\pm 0,03$ |
| $10 < d_n \leqslant 18$ | $13,3 < D_n \leqslant 23,9$ | $0,24 < r_n \leqslant 0,43$ | $\pm 0,05$ |
| $18 < d_n \leqslant 30$ | $23,9 < D_n \leqslant 40$ | $0,43 < r_n \leqslant 0,72$ | $\pm 0,09$ |

For dimensions deviating from Table C.1, the specimen can be produced with a ratio $D_n/d_n$ within the limits of 1,33 to 1,34, ratio $d_n/r_n$ within the limits of 38 to 46 and additionally with an allowance of radius $r_n \pm 12,5$ %.
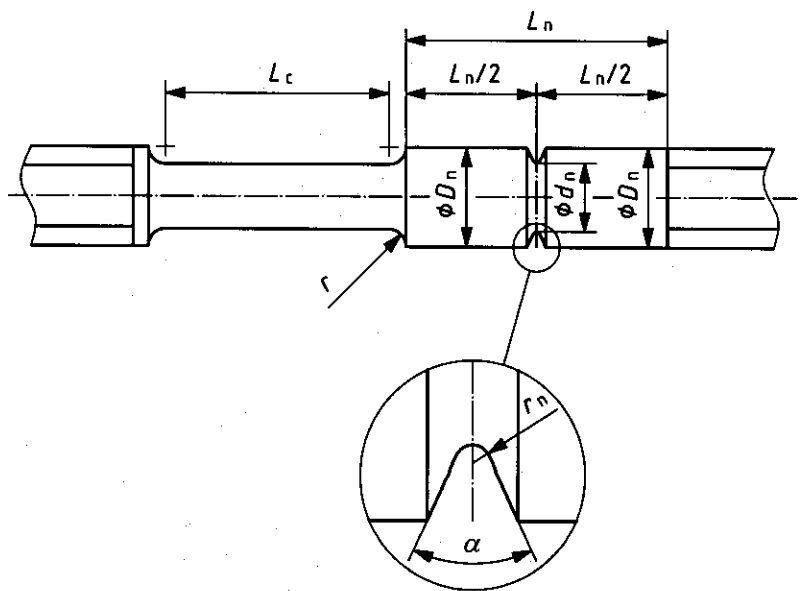
Figure C.1 — Combined notched and un-notched test piece

Earlier national standards had differences in the detailed notch geometry; however, following a research investigation carried out under the auspices of the European Creep Collaborative Committee (ECCC), it is now considered that the notch geometry, Type E, shown on Figure C.2, is suitable for assessing whether a material notch strengthens or weakens (Scholz et al.) [4].
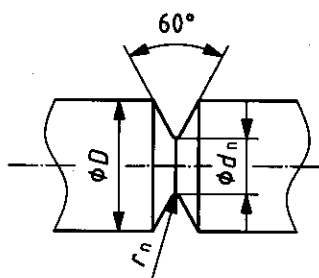


| Type | DIN | BS | E |
|------|-----|-----|---|
| $D/d_n$ | 1,25 | 1,41 | $\sqrt{1,25 \cdot 1,41} = 1,33$ |
| $D_n/r_n$ | 50 | 35 | $\sqrt{50 \cdot 35} = 42$ |

Figure C.2 — Geometry of the test pieces type DIN, BS and E

## C.3 Blunt circumferential notches

The machining of blunt circumferential notches into tensile creep test pieces is a simple cost-effective means of evaluating a material's behaviour under a multi-axial stress state, which is similar to that encountered by many industrial components under service conditions. Such notched test pieces were first advocated in 1952 by Bridgman [5]. A 'Code of Testing Practice for Creep Rupture Testing' of Bridgman notched test pieces was produced by a Working Group of the High Temperature Mechanical Testing Committee (HTMTC) in the early 1990s (Webster et al.) [6]. This latter document was subsequently revised (Webster et al.) [7], based on an EU-funded project [8].

The Code of Practice has additionally been updated to cover creep strain measurement, which may be undertaken using axial, or diametral extensometers [9]. Further information has also been published relating to diametral strain measurement on notched creep test pieces [10], [11] and calibration of diametral extensometers [12].

There is an industry-driven need to investigate the creep properties of materials over a much wider range of tri-axial tensile stress states than is provided by V notches and to give some indication of how creep strain accumulates under these circumstances. The notched bar tensile test is the most straightforward experimental procedure to achieve this aim, especially since a wide range of stress states can be generated across the notch throat by altering the notch profile. Three general classes of notch profiles are shown in Figure C.3.

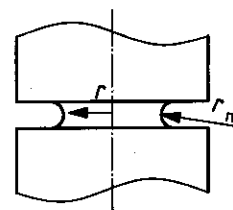The interpretation of the data generated using such notches is complex and is discussed in detail by Webster et al. [9]
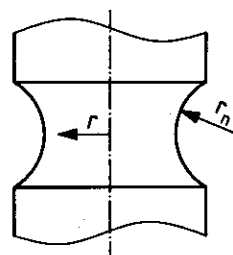


a)  Blunt
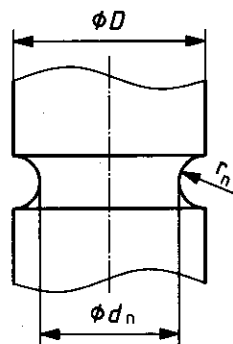


b)  Semi-circular



c)  Parallel-sided

Figure C.3 — Three possible types of Bridgman notch, Webster et al. [9]

# Annex D
## (informative)

# Method of estimating the uncertainty of the measurement in accordance with the Guide to the expression of uncertainty in measurement (GUM)

## D.1 General

Measurement uncertainty analysis is useful for identifying major sources of inconsistencies of measured results. Product standards and material property databases based on this and earlier versions of this International Standard have an inherent contribution from measurement uncertainty. It is therefore inappropriate to apply further adjustments for measurement uncertainty and thereby risk failing compliant products. For this reason, the estimates of uncertainty derived by following this procedure are for information only, unless specifically instructed otherwise by the customer.

## D.2 Introduction

This annex gives guidance on how to estimate the uncertainty of the measurements undertaken in accordance with this International Standard using a material with known creep properties. It should be noted that it is not possible to give an absolute statement of uncertainty for this test method because there are both material dependent and material independent contributions to the uncertainty statement. Hence, it is necessary to have a prior knowledge of a material's creep response to temperature and stress before being able to calculate the measurement uncertainty.

It is also shown how the estimation of measurement uncertainty may be used in conjunction with the European Creep Certified Reference Material, CRM 425, to assess compliance with this International Standard.

## D.3 Statements of uncertainty

### D.3.1 Background

Customers using accredited testing laboratories sometimes request an overall estimate of uncertainty of the accuracy of tests results. This is in accordance with the declared policy of the International Organization for Standardization (ISO) and the European standards organizations (CEN and ECISS) that all new standards concerned with testing techniques should contain a "statement of uncertainty" or a method of calculating the accuracy of the test method based upon the tolerances specified in the relevant standard. Similarly, most quality assurance systems call for an estimation of uncertainty of measurement (see EN 45001 [13] and ISO/IEC Guide 25 [14]).

In addition, two important documents have emerged from ISO Standards Committees, i.e. ISO 5725 [15] and the "*Guide to the expression of uncertainty in measurement*". Such documents largely use the terms and vocabulary given in VIM, 1993 [16] 4).

In 1995, the *Guide to the expression of uncertainty in measurement,* was published jointly by several authoritative standards bodies, namely BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML; this document is referred to hereafter as the GUM. In 2008, the GUM was reissued with minor corrections as ISO/IEC Guide 98-3 [17]. The GUM is a comprehensive document based upon rigorous statistical methods for the summation of uncertainties from various sources. Its complexity has provided the driving force for a

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4) The 1993 edition of the VIM has since been revised by ISO/IEC Guide 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*.

number of organizations to produce simplified versions of the GUM, e.g. the National Institute of Standards and Technology (NIST) in the USA (Taylor and Kuyatt [18]), the National Measurement Accreditation Service (NAMAS) in the UK (NIS 80 [19] and NIS 3003 [20]) and the British Measurement and Testing Association (BMTA [21]), also in the UK. These various documents all give guidance on how to estimate uncertainty of measurement based upon an "uncertainty budget" concept. Further information can be obtained by reference to "A Beginners Guide" (Bell [22]) and "Estimating Uncertainties in Testing" (Birch [23]). The approach adopted here for the Tensile Uncertainty Budget (Loveday [24]) is similar to that proposed for a creep testing uncertainty budget used in association with the Creep Certified Reference Material, CRM 425 (Loveday [25]). Comprehensive statements of uncertainty have also now been published as part of the EU-funded project 'Uncert' (Kandil et al. [26]) and an additional document has now been issued covering creep uncertainty as a CEN-endorsed Technical Workshop Agreement, CWA 15261-2 [27]

The following analysis is a simplified method for estimating uncertainty in creep testing, based upon the concepts given in the GUM, shown schematically in Figure D.1. The total uncertainty of a measurement is determined by summing all the contributing components in an appropriate manner. It is necessary to quantify all the contributions, and, at the preliminary evaluation stage, to decide whether some contributions are negligible and therefore not worth including in the subsequent calculations. For most practical measurements in the materials field, the definition of negligible may be taken as a component smaller than one-fifth of the largest component. The GUM categorizes two ways of evaluating uncertainties, Type A and type B. Type A determination is by repeat observations and, provided sufficient readings are available, e.g. more than nine, conventional statistical analysis can be used to determine the standard deviations.
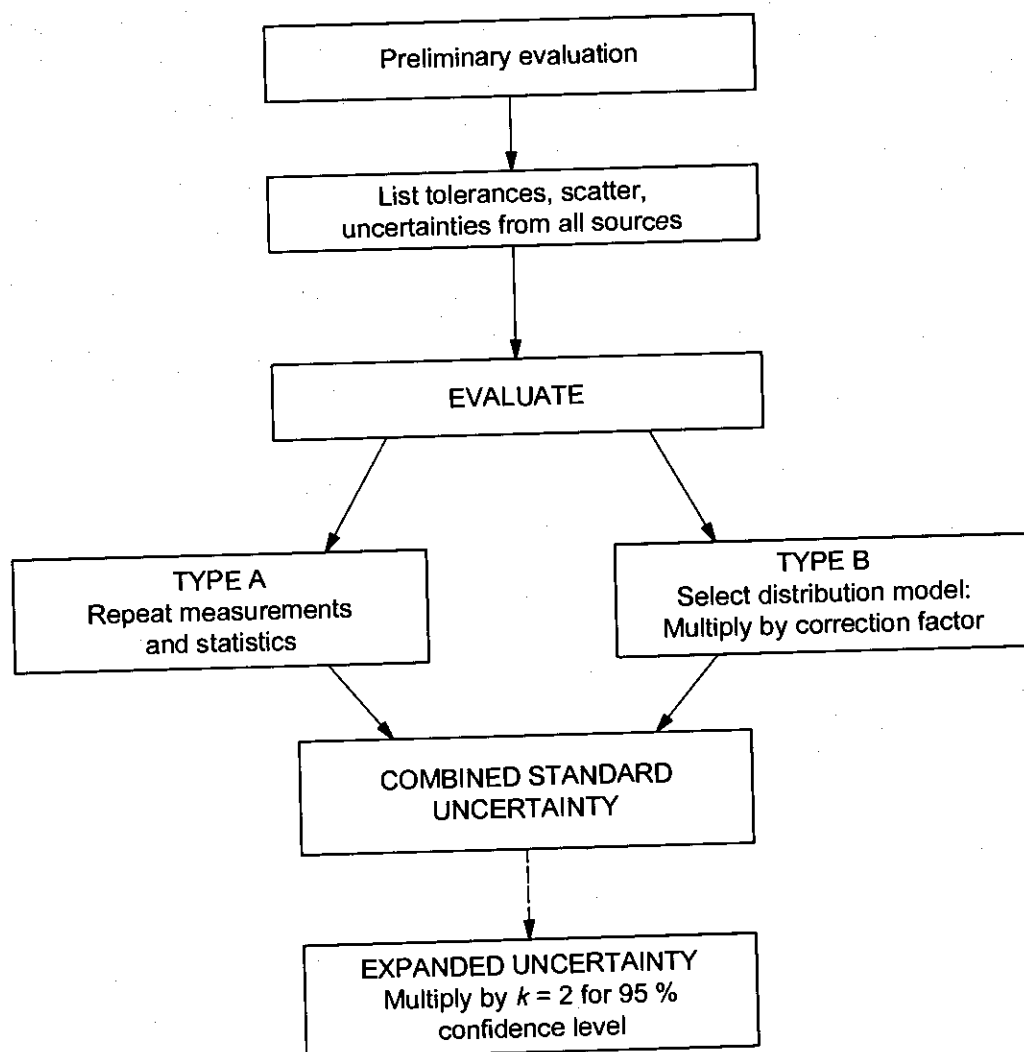


Figure D.1 — Outline procedure for estimation of uncertainty

Type B evaluation is by means other than Type A and makes use of, for example, tolerances specified in standards, measured data, manufacturer's specifications, calibration certificates and in most cases a knowledge of a simple model of the relationship between the various components, and of the likely distribution model of the components. If, for example, the tolerance specified in a standard is $\pm a$, then in absence of any other knowledge, it may be appropriate to assume a rectangular distribution model, in which case the uncertainty becomes $u_s = a/\sqrt{3}$ .

If better knowledge is available, it may be that a triangular distribution is more appropriate, in which case $u_s = a/\sqrt{6}$ , (see the GUM). The next step is to determine the combined standard uncertainty, $u_c$, by summing the standard uncertainties, usually by using the root sum square method. The expanded uncertainty, $U_E$, is then obtained by multiplying $u_c$ by a coverage factor, $k$, where $k = 2$ for a 95 % confidence level; thus, $U_E = 2u_c$. This procedure is shown schematically in Figure D.2.
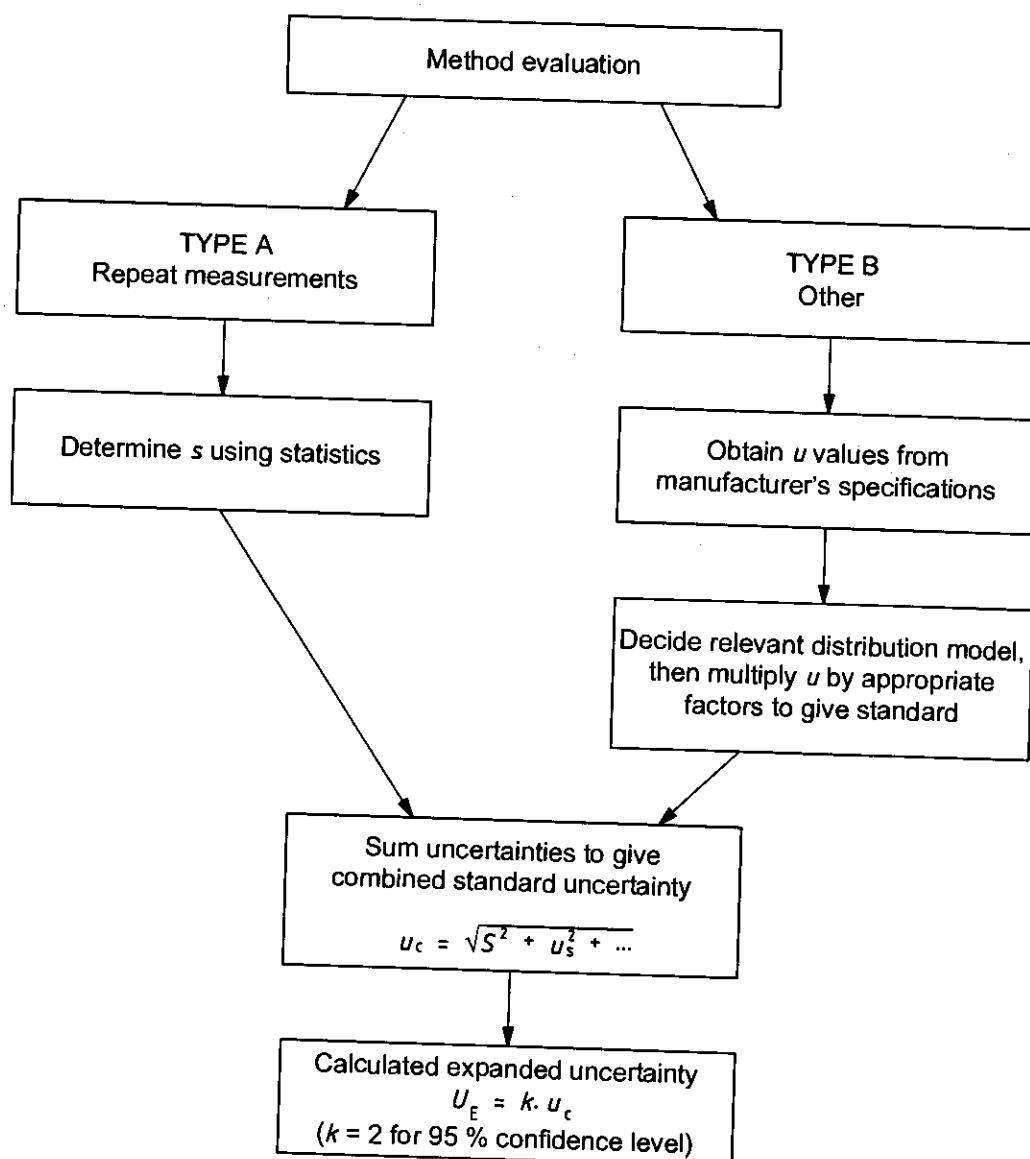


Figure D.2 — Detailed procedure for estimating uncertainty in accordance with the GUM

### D.3.2 Statement of uncertainty: creep testing

In the case of most metallic materials, over a limited stress range the minimum creep rate, $\dot{\varepsilon}_{min}$, may be related to the applied stress, $\sigma$, and the temperature, $T$, by a relationship having the form:

$$\dot{\varepsilon}_{min} = A\,\sigma^n \exp\left(-Q/RT\right) \tag{D.1}$$

where

$A$    is a material constant;

$n$    is the stress index in the Norton Creep Law;

$Q$    is the activation energy;

$R$    is the gas constant.

Since for most materials, to a first approximation, the creep rupture time, $t_u$, is directly proportional to the inverse of the minimum creep rate, it can be seen that errors in $t_u$ and $\dot{\varepsilon}_{min}$ are due to errors in $\sigma$ and $T$ in the two separate components of Equation (D.1). Tolerances for $\sigma$ and $T$ are specified in testing standards; however, the parameters $n$ and $Q$ are material dependent. Thus it is not possible to quote an overall uncertainty value applicable for all materials which are tested in accordance with the standard.

Using Equation (D.1), it has been shown elsewhere (Loveday [25]) that for the solid solution nickel base alloy, Nimonic 75, with a creep activation energy, $Q = 345\,kJ\,mol^{-1}$, a stress index $n = 6$, together with the temperature and stress tolerances as permitted in this International Standard, the expanded measurement uncertainty $U_E = 20,2\,\%$ at the 95 % confidence level.

Similarly, Granacher and Holdsworth [28] have compiled uncertainty budgets including a contribution to the overall uncertainty due to the precision of the strain measurement system specifically for assessing the measurement uncertainties for the times to achieve 0,2 % and 1 % plastic strains for interrupted and uninterrupted tests. The materials examined included two ferritic steels (2¼Cr-1Mo at 500 °C, and 1Cr-1Mo-0,5 Ni-0,25V at 550 °C), one martensitic steel (12Cr-1Mo-0,3V at 600 °C) and one austenitic steel (17Cr-13Ni-2Mo-0,2N at 600 °C) and times typically in the range of 30 000 hours. A summary of their estimates of the measurement uncertainties treating the tolerances as rectangular distributions and expressed at the 95 % confidence level in accordance with the GUM are given in Table D.1

**Table D.1 — Range of uncertainties for $t_{p0,2}$ and $t_{p1}$**

| Interrupted tests % | Uninterrupted tests % |
|---|---|
| 27 to 38 | 27 to 32 |

It should be noted that in addition there are other factors that can affect the measurement of creep properties such as test piece bending, or methods of gripping the test piece, etc. However, since there is insufficient quantitative data available on these effects, it is not possible to include their influence in uncertainty budgets at present. It should also be recognized that this uncertainty budget approach only gives an estimate of the uncertainty due to the measurement technique and does not make an allowance for the inherent scatter in experimental results attributable to material inhomogeneity.

The uncertainty budget presented here could be regarded as an upper bound to the measurement uncertainty for a laboratory undertaking testing in compliance with this International Standard.

## D.4 A reference material for creep testing

### D.4.1 General

During recent years, the benefits of the use of Certified Reference Materials in the field of mechanical testing have been recognized. Under the auspices of the Community Bureau of Reference (BCR), a reference material has been developed for creep testing (Gould and Loveday [29]); see Table D.2.

Table D.2 — Certified values for the Nimonic 75 Creep Reference Material, CRM 425

| Property [a] | Certified value [b] | Uncertainty [c] |
|---|---|---|
| Creep rate at 400 h | $71,8 \times 10^{-6}$ h$^{-1}$ | $5 \times 10^{-6}$ h$^{-1}$ |
| $t_{p2}$ | 278 h | 16 h |
| $t_{p4}$ | 557 h | 30 h |
| [a] Testing conditions: $T = 600$ °C, $\sigma_0 = 160$ MPa. | | |
| [b] This value is the unweighted mean of the means of the results from 9 laboratories each of which made 5 separate determinations of the certified property. | | |
| [c] The uncertainty is taken as half the 95 % confidence interval of the mean defined in b. | | |

The CRM 425 is available from BCR Reference Materials, (Community Bureau of Reference), Management of Reference Materials (MRM) Unit, Joint Research Centre, Institute for Reference Materials and Measurement (IRMM), Retieseweg, B-2440, Geel, Belgium.

### D.4.2 Using the CRM 425 for assessing uncertainty

For the Nimonic 75 CRM, a test undertaken in accordance with this International Standard at 600 °C has a permissible temperature tolerance of ± 3 °C, and allowing for the tolerance on the measurement of stress (± 1 %), the expected total uncertainty is ~ 20,2 % calculated in accordance with the GUM (see D.3.2). If the tolerance due to testing is added to the uncertainty of the certified value using a root sum square approach, then it is possible to calculate the total error band within which data from a single test may be expected to lie, as shown in Table D.3.

Table D.3 — Acceptable data range for creep testing using the Creep Reference Material, CRM 425

| Parameter | Certified value | Uncertainty 95 % confidence level | Testing [a] tolerance (± 20,2 %) | Total uncertainty ~ 21 % | |
|---|---|---|---|---|---|
| | | | | Value | Range |
| Creep rate at 400 h ($10^{-6}$ h$^{-1}$) | 72 | 5 | ± 14,5 | ± 15,3 | 56,7 to 87,3 |
| $t_{p2}$ (h) | 278 | 16 | ± 56,2 | ± 58,4 | 219,6 to 336,4 |
| $t_{p4}$ (h) | 557 | 30 | ± 112,5 | ± 116,4 | 440,6 to 673,4 |
| [a] Assuming $\Delta T = \pm 3$ °C, $\Delta\sigma = 1$ %, stress index $n = 6$ and creep activation energy $Q = 345$ kJ mol$^{-1}$. | | | | | |

## D.5 Uncertainties in creep testing of single crystal nickel-base superalloy at 1 100 °C

There is a need for an operation of advanced gas turbines at an ultra-high temperature. The creep properties of materials used in the gas turbines need to be evaluated and verified at high temperatures. This means it is important to establish a creep testing method for application at temperatures above 1 000 °C.

In order to establish a testing method for creep rupture properties of superalloys at temperatures above 1 000 °C, a Round Robin test (RRT) was carried out under the programme set up by the Standardization Committee on High Temperature Creep and Creep Rupture Testing at the New Materials Center (NMC). Nine groups of research institutes and companies participated in the programme. The samples tested were of new Ni-base single-crystal superalloy (designated name: TMS-82+; see Table D.4), developed in the High Temperature Materials 21 Project at NIMS. Three repeat creep rupture tests of TMS-82+ were carried out at five laboratories under test conditions of 137 MPa and 1 100 °C (see Table D.5). The previously reported rupture time under these test conditions is 340 h. The evaluation of uncertainties in the determination of the results of creep test at 1 100 °C was carried out according to the GUM. Guidelines for characterizing the creep and creep rupture properties of single crystal superalloy at temperatures above 1 000 °C were derived from the RRT reported elsewhere (see References [30], [31] and [32]).

### Table D.4 — Chemical composition of tested alloy (mass %)

| Material | Co | Cr | Mo | W | Al | Ti | Ta | Hf | Re | Ni |
|---|---|---|---|---|---|---|---|---|---|---|
| TMS-82+ | 7,8 | 4,9 | 1,9 | 8,7 | 5,3 | 0,5 | 6,0 | 0,1 | 2,4 | Balance |

Solution treatment 1 300 °C, 1h →1 320 °C, 5h Ar Gas Fan Cool

Two-step aging treatment 1 100 °C, 4h Ar GFC 870 °C, 20h Ar Gas Fan Cool

### Table D.5 — Summary of the creep rupture tests reported by five laboratories

### TMS-82+, 137 MPa and 1 100 °C

| Properties | $n$ | Data range | Average |
|---|---|---|---|
| Time to rupture | 19 | 238,6 ~ 460,8 (h) | 333,9 (h) |
| Elongation | 19 | 6,3 ~ 13,4 (%) | 10,3 (%) |
| Reduction of area | 19 | 24,7 ~ 38,9 (%) | 33,7 (%) |

To obtain the usual 95 % confidence level, a coverage factor of 2 should be applied to the standard uncertainties.

Ni-base single crystal superalloy (TMS-82+) at 1 100 °C and 137 MPa

| Time to rupture | (334 ± 59) h |
| Elongation | (10,0 ± 5,2) % |
| Reduction of area | (34,0 ± 8,2) % |

# Annex E
## (informative)

# Representation of results and graphical extrapolation

## E.1 General

This annex summarizes important information, which should help the user to apply the established methodology developed within the European Creep Collaborative Committee [33].

## E.2 Symbols for strength values and their calculation

### E.2.1 Strain

With the exception of the percentage elongation after creep rupture ($A_u$), the symbol $\varepsilon$ is used for the strain.

In most cases, the anelastic strain, $\varepsilon_k$, is negligible and there is no difference between the plastic strain, $\varepsilon_p$, and the permanent strain, $\varepsilon_{per}$.

### E.2.2 Creep rupture strength

The creep rupture strength at a specified test temperature, $T$, is the applied stress, $\sigma_o$, which leads to rupture after a certain test duration (creep rupture time, $t_u$) under constant tension force.

For the creep rupture strength, the symbol $R_u$ is used, followed by the second index for the creep rupture time, $t_u$, in hours, and by the third index for the test temperature, $T$, in degrees Celsius (°C).

EXAMPLE    For the short symbol of the creep rupture strength determined at a creep rupture time of $t_u = 100\ 000$ h and a test temperature of $T = 550$ °C (100 000 h-creep-rupture strength at 550 °C):

$$R_{u\ 100\ 000/550}$$

### E.2.3 Stress-to-specific-plastic-strain

The stress-to-specific-plastic-strain is the applied stress, $\sigma_o$, at a specified test temperature, $T$, which leads to a predetermined plastic strain, $x$, after a certain test duration (time-to-specific-plastic-strain, $t_{px}$) under constant load.

For the stress-to-specific-plastic-strain, the symbol $R_p$ is used, followed by the second index for the maximum value of the plastic strain, $x$, in percent, by the third index for the time-to-strain value and by the fourth index for the test temperature.

EXAMPLE    For the short symbol of the stress-to-specific-plastic-strain, with a maximum value of plastic strain of 0,2 %, a time-to-strain value of 1 000 h and a test temperature of $T = 650$ °C:

$$R_{p\ 0,2\ 1\ 000/650}$$

## E.3 Specimens

### E.3.1 Shape and dimension of smooth specimens

To avoid rupture position near to the end of gauge length, it is recommended to exploit half the shape tolerance for a tapering of the specimen up to the centre of the gauge length.

### E.3.2 Shape and dimension of notched specimens

Normally specimens with circular cross-sections shall be used. The use of shapes and dimensions of specimens with non-circular cross-sections must be declared.

If not otherwise expressly agreed, notched specimens with circular cross-sections should feature a notch geometry according to Figure E.1. The length of the shaft with diameter $D_n$ should be at least equal to the root diameter, $d_n$.

**Table E.1 — Examples for dimensions of notched specimens with circular cross-sections and with an elastic stress concentration factor $K_t = 4,5 \pm 0,5$**

| root diameter, $d_n$, in mm ± 0,01 mm | 6 | 8 | 10 | 12 |
|---|---|---|---|---|
| shaft diameter, $D_n$, in mm ± 0,1 mm | 8 | 10,6 | 13,3 | 16 |
| notch radius, $r_n$, in mm | 0,14 | 0,20 | 0,25 | 0,3 |
| allowance of radius, $r_n$, in mm | ± 0,02 | ± 0,03 | ± 0,04 | ± 0,04 |

For dimensions deviating from Table E.1 the specimen can be accomplished with a ratio $D_n/d_n$ within the limits of 1,33 to 1,34, ratio $d_n/r_n$ within the limits of 38 to 46 and additionally with an allowance of radius $r_n$ of ± 12,5 %.

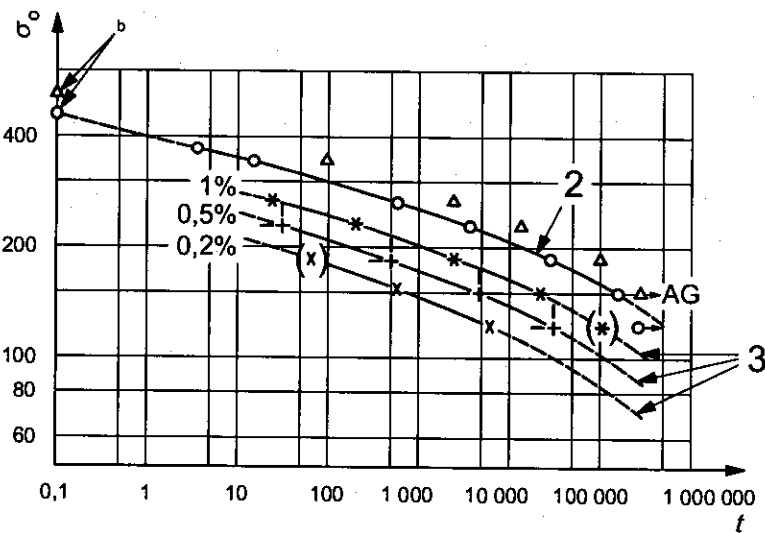The elastic stress concentration factor [3] is calculated using Equation (E.1):

$$K_t = 1 + \left( \frac{1}{2} \cdot \frac{r_n / d_n}{D_n / d_n - 1} + \frac{r_n}{d_n} \cdot \left( 1 + 2 \cdot \frac{r_n}{d_n} \right)^2 \right)^{-1/2} \qquad (E.1)$$
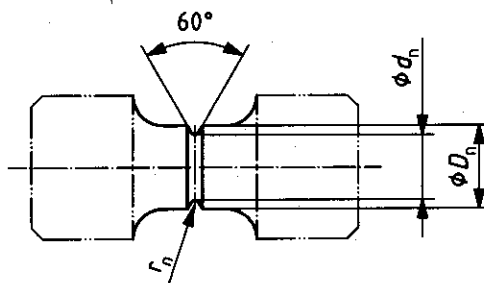


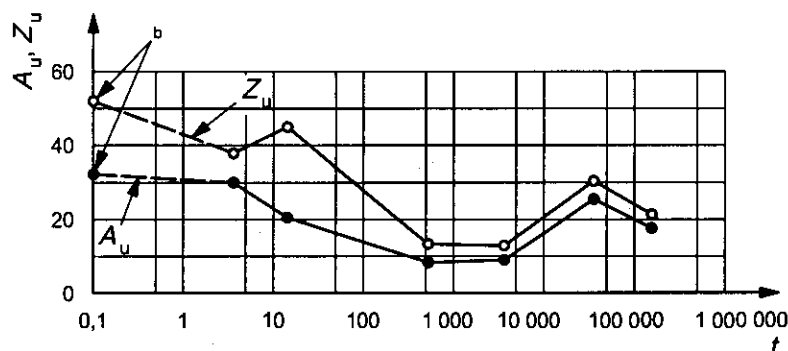**Figure E.1 — Schematic diagram of a notched specimen with a circular cross-section**
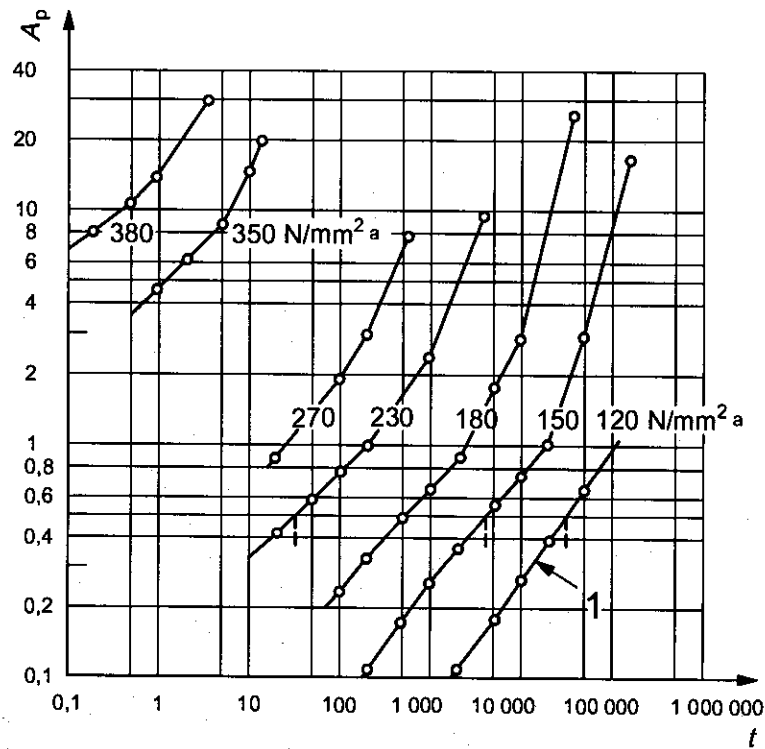
**a)   Creep diagram**



**b)   Creep rupture diagram**

**Figure E.2** (*continued*)

c) Creep rupture deformation diagram

**Key**

| | | | |
|---|---|---|---|
| 1 | creep curve | o→AG | test stopped without rupture |
| 2 | creep rupture curve | o→ | test running |
| 3 | creep rupture deformation diagram | △→AG | test stopped without rupture |
| o | smooth test pieces (rupture) | △→ | test running |
| △ | notched test pieces (rupture) | – – | extrapolated |

a    Initial stress.

b    Hot tensile test.

**Figure E.2 — Example for the representation of test results for constant test temperature and constant tractive force**

## E.4 Evaluation

### E.4.1 General

The experimental results of an individual material for one test temperature can be displayed and evaluated in a number of diagrams (see Figures E.2 and E.3). In these diagrams, extrapolated curves should be dashed while extrapolated points should be in parentheses. In E.5, some remarks about the extrapolation of data are given.
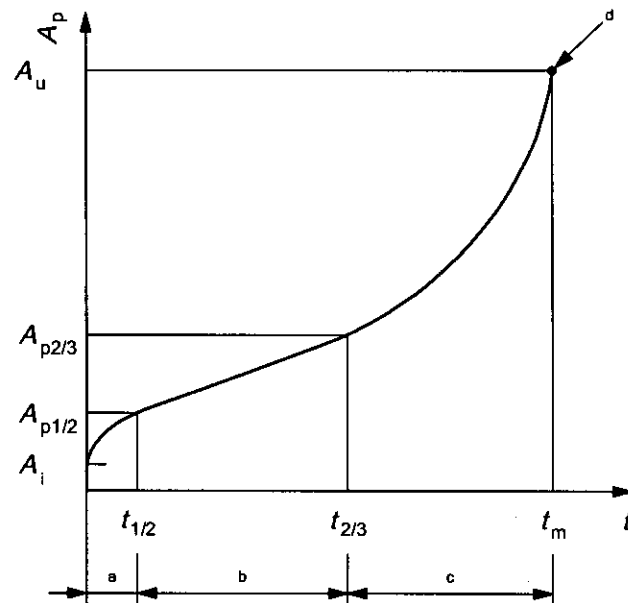
**Key**

a    Primary creep stage.

b    Secondary creep stage.

c    Tertiary creep stage.

d    Rupture.

**Figure E.3 — Linear creep diagram (schematic)**

## E.4.2 Logarithmic creep diagram

In order to display creep curves, the plastic strain, $\varepsilon_p$, will be plotted versus time, $t$, in a diagram with both axes in logarithmic scales [see Figure E.2 a)].

The creep curve can either be displayed smooth or as a series of lines connecting the measured data. The time to specific plastic strain, $t_{px}$, can be taken from such a diagram.

## E.4.3 Creep rupture diagram

To determine the creep strain diagram, the times to strain corresponding to given strain values, e.g. $t_{p0.2}$, will be plotted in dependence of initial stress, $\sigma_0$, in logarithmic scales [see Figure E.2 b)]. The curve should be smooth. From this diagram, the stress-to-strain, $R_{x,t,T}$, is taken.

To determine the creep rupture diagram, the rupture time, $t_u$, will be plotted in dependence of initial stress, $\sigma_0$, in the same diagram and smoothened.

From this curve, the stress-to-rupture, $R_{u,t,T}$, is taken.

The rupture strength and the stresses-to-strain from hot tensile tests can be depicted in this diagram at a certain time, e.g. $t = 0,1$ h. In this case it has to be properly denoted in the figure.

Furthermore, the rupture times depending on the initial stress, $\sigma_0$, of notched specimens can be plotted as a hint in this diagram. Additional judgments of the material behaviour can be achieved in this way.

### E.4.4 Creep rupture elongation diagram

In this diagram the values of creep rupture strain, $A_u$, and reduction of area after creep rupture, $Z_u$, are plotted versus the logarithm of creep rupture time, $t_u$.

Rupture strain and reduction of area from hot tensile tests can be plotted in this diagram as a hint at a certain time, e.g. $t = 0,1$ h. In this case it has to be properly denoted in the figure.

### E.4.5 Creep diagram with linear scales

In order to display creep curves, the plastic strain, $\varepsilon_p$, will be plotted versus time, $t$, in a diagram with both axes in linear scales (see Figure E.3). The creep curve can either be displayed smoothened or as a series of lines connecting the measured data.

From the slope of this curve the creep rate, $\dot{\varepsilon}$, depending on time, $t$, can be determined as well as the overall minimum creep rate, $\dot{\varepsilon}_{p,min}$. The transition times $t_{12}$ and $t_{23}$ which mark the transition from the primary to secondary creep stage ($t_{12}$) and from secondary to tertiary creep ($t_{23}$) can be taken from this diagram. Not all linear creep curves display distinguished stages of creep 1, 2 and 3.

## E.5 Extrapolation

### E.5.1 General

During the evaluation of creep data it is often necessary to determine values of creep rupture strength or stress-to-strain which exceed the longest experimental time by a factor $q_e$. This factor $q_e$ is known as the extrapolation-time-ratio and should not be greater than 3.

It is recommended to always denote the extrapolation-time-ratio, $q_e$, and to indicate if the extrapolated creep strength falls below the minimum initial stress level, $\sigma_{omin}$, for the same material. In this case extrapolation uncertainties are usually large.

Changes in the microstructure or creep rupture deformation values depending on time and/or test temperature should be taken into consideration during extrapolation. The extrapolation procedure should be denoted.

### E.5.2 Graphical extrapolation and creep rupture diagram

Often the extrapolation is carried out as graphical prolongation of the creep rupture curve and/or the creep stress curve(s). The transient of adjacent curves at the same test temperature [see Figure E.4 b)] or comparable curves at different, preferably higher test temperatures [see Figure E.4 c)], can be used as hints for the extrapolation. The same can be achieved from the prolongation of creep strain curves. More advice is available from ECCC [33].

If graphical extrapolation has been carried out with the help of adjacent curves, their smaller extrapolation-time-ratio, $q_e$, can be denoted [see Figure E.4 b) or Figure E.4 c)].

### E.5.3 Extrapolation by means of time-temperature-parameters

Often the logarithm of stress, $\sigma_o$, is plotted versus a time-temperature-parameter which is derived from the test temperature and the creep rupture time or time-to-strain. The data points are fitted by a so-called "master curve".

It is recommended to use optimized time-temperature-parameters whose dependence on time and test temperature is fitted to test results. Furthermore, tests with longer durations should be given greater weight in the curve fitting process. It should be mentioned that small scatter of data does not guarantee the accuracy of an extrapolation.

For given values of creep rupture strength or time-to-strain, which have been taken from the master curve respectively, extrapolations can be carried out for given test temperatures. In order to improve the quality of extrapolation the extrapolated values should be plotted in the creep strain (rupture) diagram and compared to the measured values. Further advice for evaluation and extrapolation can be found in Reference [34].

## E.6 Test report, recommended additional information

In the test report, it is recommended to include the following additional information:

— information regarding the material of the sample;

— material and material number according to Reference [37];

— manufacturer;

— cast number, cast weight;

— steel making process, production process;

— block weight, piece weight;

— characteristic measurements, form of semi-manufacture;

— location of the sample in the block/piece;

— chemical composition, heat treatment;

— results of tensile test(s) at room temperature;

— impact test data (Charpy, Izod, etc.);

— results of tensile test(s) at elevated temperature(s);

— microstructure;

— when appropriate: extrapolation procedure and extrapolation-time-ratio.
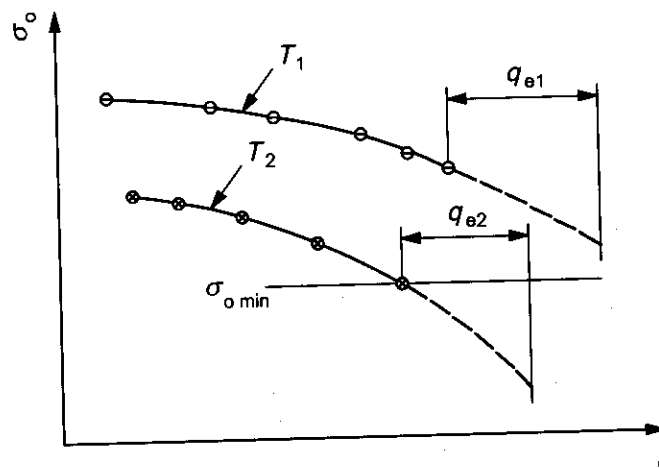
a) **Creep rupture curves,** $T_1 < T_2$
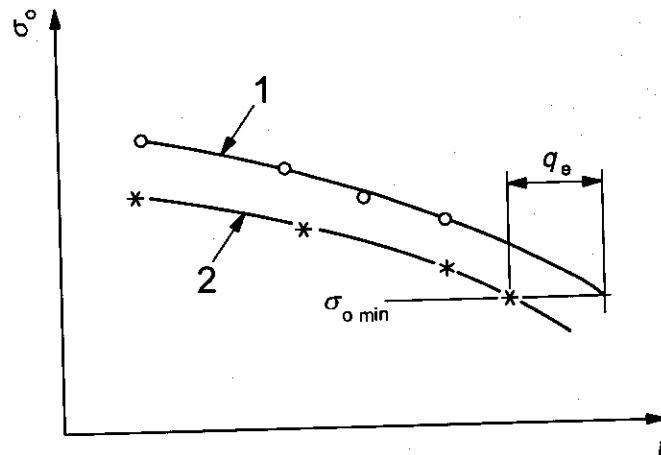
b) **Creep rupture and creep strain curves,** $T = $ constant
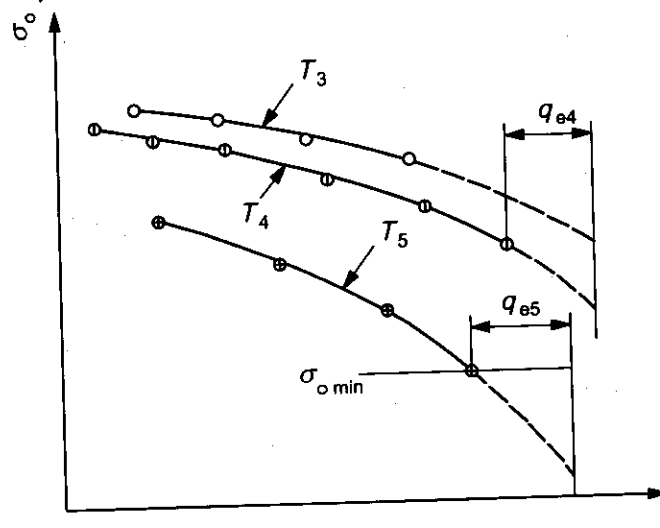
c) **Creep rupture curves,** $T_3 < T_4 \ll T_5$

**Key**

1   creep rupture curve

2   time to strain curve

**Figure E.4 — Exemplary extrapolation cases in creep strain (rupture) diagram**

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