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

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Foreword

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

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 204:2009), which has been technically revised. The main changes compared to the previous edition are as follows:

— Some of the symbols have been changed to achieve harmonization with the ISO 6892 series.

— For the purpose of this document, the terms “fracture” and “rupture” are interchangeable.

— The term “indicated temperature”, T_i, has been replaced by “corrected measured temperature”, T_c, with errors from all sources being taken into account and any systematic errors having been corrected. The terms “elongation” and “extension” have been clarified and aligned with the terms used in the ISO 6892 series. Elongation refers to the test piece deformation measured manually either during deliberate test interruptions or after fracture, whilst extension is determined by continuous measurement using an extensometer.

— Some information relating to the calibration of thermocouples has been transferred from an informative annex into the main body of the document.

— Some changes have been made to Table 1 and formulae have been amended using reference length, L_r.

— Equation E.1 (now Formula C.1) has been corrected.

— A new informative annex relating to computer compatible representation of standards has been added.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Creep is the phenomenon exhibited by materials which slowly deform when subjected to loading at elevated temperature. This document is concerned with the method used to measure such material behaviour.

Annexes are included concerning temperature measurement using thermocouples and their calibration, creep testing test pieces with circumferential V and blunt (Bridgman) notches, estimation of measurement uncertainty, methods of extrapolation of creep rupture life and information about computer compatible representation of standards.

NOTE 1 Information is still sought relating to the influence of off-axis loading or bending on the creep properties of various materials. Based on the future availability of quantitative data, consideration might be given 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[43].

NOTE 2 Information concerning the benefit of standards being produced in a computer compatible format is given in Annex F.

This document incorporates many recommendations developed through the European Creep Collaborative Committee (ECCC).

NOTE 3 Several different gauge lengths and reference lengths are specified in this document. 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 can 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 3.3). “Extension” is used for uninterrupted creep test with continuous measurement of the increase of the length of the test piece by using an extensometer. “Elongation” is mainly used for interrupted creep test with the manual measurement of the increase of the length of the test piece.

NOTE 4 For many applications, the term “strain” is synonymous with extension.

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

1 Scope

This document specifies the methods for

a) uninterrupted creep tests with continuous monitoring of extension,

b) interrupted creep tests with periodic measurement of elongation,

c) stress rupture tests where normally only the time to fracture is measured,

d) a test to verify that a predetermined time can be exceeded under a given force, with the elongation or extension not necessarily being reported.

NOTE A creep test can be continued until fracture has occurred or it can be stopped before fracture.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6892-1, *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

ISO 6892-2, *Metallic materials — Tensile testing — Part 2: Method of test 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 extensometer systems used in uniaxial testing*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
reference length
L_r
base length used for the calculation of either percentage elongation or percentage extension

Note 1 to entry: A method to calculate this value is given in 7.5.

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 to entry: In general, $L_o \geq 5D$.

3.3
extensometer gauge length
L_e
distance between the measuring points of the extensometer

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

3.5
final gauge length after fracture
L_u
length between gauge length marks on the test piece measured after fracture, at ambient temperature, with the pieces carefully fitted back together with their axes in a straight line

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

3.8
initial stress
R_o
applied force divided by the original cross-section area, S_o, of the test piece

3.9
extension
ΔL_e
increase of extensometer gauge length, L_e, at time t and at test temperature

Note 1 to entry: For further information, see 6.2.

3.10
elongation
ΔL_o
increase of original gauge length, L_o, at time t

Note 1 to entry: For further information, see 6.2.

3.11
percentage extension
e
extension at test temperature expressed as a percentage of the reference length, L_r, as given in Formula (1)

$$e = \frac{\Delta L_e}{L_r} \times 100 \quad (1)$$

Note 1 to entry: See Figure 1.

3.12
percentage elongation
A
elongation expressed as a percentage of the reference length, L_r, as given in Formula (2)

$$A = \frac{\Delta L_o}{L_r} \times 100 \quad (2)$$

3.13
percentage elastic extension
e_e
extension at test temperature expressed as a percentage of the reference length, L_r, which is proportional to the initial stress, R_o

Note 1 to entry: This value can be calculated from the stress/percentage extension values during loading. See 8.4.2.

Note 2 to entry: See Figure 1.

3.14
percentage initial total extension
e_{ti}
extension at test temperature expressed as a percentage of the reference length, L_r, at end of loading with the initial stress, R_o

Note 1 to entry: See Figure 1.

3.15
percentage initial plastic extension
e_i
extension at end of loading and at test temperature with the initial stress, R_o, expressed as a percentage of the reference length, L_r, and determined as the difference between the percentage initial total extension, e_{ti}, and the percentage elastic extension, e_e, as given in Formula (3)

$$e_i = e_{ti} - e_e \quad (3)$$

Note 1 to entry: See Figure 1.

Note 2 to entry: This value represents the plastic extension during the loading phase.

3.16
percentage total extension
e_t
extension at the test force at time *t* and at test temperature, expressed as a percentage of the reference length, L_r

Note 1 to entry: See Figure 1.

3.17
percentage plastic extension
e_p
extension at time *t* and at test temperature determined as the difference between the percentage total extension, e_t, and the percentage elastic extension, e_e, expressed as a percentage of the reference length, L_r, as given in Formula (4)

$$e_p = e_t - e_e \quad (4)$$

Note 1 to entry: See Figure 1.

3.18
percentage total ultimate extension
e_u
total extension at rupture and at test temperature, expressed as a percentage of the reference length, L_r

3.19
percentage creep extension
e_f
extension at loading determined and at test temperature as the difference between the percentage plastic extension, e_p, and the percentage initial plastic extension, e_i, expressed as a percentage of the reference length, L_r, as given in Formula (5)

$$e_f = e_p - e_i \quad (5)$$

Note 1 to entry: See Figure 1.

Note 2 to entry: Suffix f originates from “fluage”, “creep” in French.

3.20
percentage anelastic extension
e_k
negative extension at end of unloading at test temperature, expressed as a percentage of the reference length, L_r

Note 1 to entry: See Figure 1 and 8.4.

3.21
percentage permanent extension
e_{per}
extension at end of unloading and at test temperature determined as the difference between the percentage total extension, e_t, and the sum of percentage elastic extension, e_e, plus the percentage anelastic extension, e_k, expressed as a percentage of the reference length, L_r, as given in Formula (6)

$$e_{per} = e_t - (e_e + e_k) \quad (6)$$

Note 1 to entry: In the case of $e_k \approx 0$, the following relationship may be used: $e_{per} \approx e_p$.

Note 2 to entry: See Figure 1.

3.22
percentage permanent elongation
A_{per}
elongation expressed as a percentage of the reference length, L_r, at end of unloading and at room temperature

3.23
percentage elongation after creep fracture
A_u
permanent elongation after fracture, $L_u - L_o$, expressed as a percentage of the reference length, L_r, as given in Formula (7)

$$A_u = \frac{L_u - L_o}{L_r} \times 100 \quad (7)$$

Note 1 to entry: A_u may have the specified temperature, T, in degrees Celsius as superscript and the initial stress, R_o, in megapascals as subscript; see the example in Table 1.

3.24
percentage reduction of area after creep fracture
Z_u
maximum change in cross-sectional area measured after fracture, $S_o - S_u$, expressed as a percentage of the original cross-sectional area, S_o, as given in Formula (8)

$$Z_u = \frac{S_o - S_u}{S_o} \times 100 \tag{8}$$

Note 1 to entry: Z_u may have the specified temperature, T, in degrees Celsius as superscript and the initial stress, R_o, in megapascals as subscript; see the example in Table 1.

3.25
creep extension time
t_{fx}
time required for a strained test piece to obtain a specified percentage creep extension, x, at the specified temperature, T, and the initial stress, R_o

EXAMPLE $t_{f0,2}$

3.26
plastic extension time
t_{px}
time required to obtain a specified percentage plastic extension, x, at the specified temperature, T, and the initial stress, R_o

Note 1 to entry: An example for t_{p1} is given in Figure E.2 a) (t_{p1} = 100 000 h corresponds to e_p = 1 % at R_o = 120 MPa).

3.27
creep rupture time
t_u
time to rupture for a test piece maintained at the specified temperature, T, and the initial stress, R_o

Note 1 to entry: The symbol t_u can have as superscript the specified temperature, T, in degrees Celsius and as subscript the initial stress, R_o, in megapascals; see the example in Table 1.

3.28
single test piece machine
testing machine that permits straining of a single test piece

3.29
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	Designation
D	mm	Diameter of gauge 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/unnotched test piece (see Figure C.1)
d_n	mm	Diameter across root of circumferential notch For a combined notched/unnotched 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)]
ΔL_{et}		Increase of extensometer gauge length at time t
ΔL_{ot}	mm	Increase of original gauge 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 fracture
L_c	mm	Parallel length
L_e	mm	Extensometer gauge length
r_t	mm	Transition radius
r_n	mm	Notch root radius
S_o	mm^2	Original cross-sectional area of the parallel length
S_u	mm^2	Minimum cross-sectional area after fracture
R_o	MPa	Initial stress
e	%	Percentage extension
e_e	%	Percentage elastic extension
e_f	%	Percentage creep extension: $e_f = \frac{\Delta L_{ot}}{L_r} \times 100$ NOTE As an example, the symbol can be completed as follows: $e_{f50/5\,000}^{375}$: percentage creep extension with an initial stress of 50 MPa after 5 000 h at the specified temperature of 375 °C.
e_{fu}	%	Percentage creep extension at creep rupture time
e_i	%	Percentage initial plastic extension
e_k	%	Percentage anelastic extension

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).

NOTE For the purposes of creep testing in this document, the terms "fracture" and "rupture" are interchangeable and are used to describe when a test piece breaks.

Table 1 *(continued)*

Symbol[a]	Unit	Designation
e_p	%	Percentage plastic extension
e_{per}	%	Percentage permanent extension
e_{pu}	%	Percentage plastic extension at creep rupture time
e_t	%	Percentage total extension
e_u	%	Percentage total extension at creep rupture time
A_{per}	%	Percentage permanent elongation NOTE As an example, the symbol can be completed as follows: $A^{375}_{per50/5\,000}$: percentage permanent elongation with an initial stress of 50 MPa after 5 000 h at the specified temperature of 375 °C.
A_u	%	Percentage elongation after creep fracture: $A_u = \frac{L_u - L_o}{L_r} \times 100$ NOTE As an example, the symbol can be completed as follows: $A^{375}_{u\,50}$: percentage elongation after creep fracture with an initial stress of 50 MPa at the specified temperature of 375 °C.
Z_u	%	Percentage reduction of area after creep fracture: $Z_u = \frac{S_o - S_u}{S_o} \times 100$ NOTE As an example, the symbol can be completed as follows: $Z^{375}_{u\,50}$: percentage reduction of area after creep fracture with an initial stress of 50 MPa at the specified temperature of 375 °C.
t	h	Elapsed time from end of loading
t_{fx}	h	Creep extension time
t_{px}	h	Plastic extension time
t_u	h	Creep rupture time NOTE As an example, the symbol can be completed as follows: $t^{375}_{u\,50}$: 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_c	°C	Corrected measured temperature
x	%	Specified percentage creep or plastic extension
n		Norton 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).

NOTE For the purposes of creep testing in this document, the terms “fracture” and “rupture” are interchangeable and are used to describe when a test piece breaks.

5 Principle

The test consists of heating a test piece to the specified temperature and of straining it 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 extension (uninterrupted test) with continuous extension measurement;

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

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

NOTE "Constant stress" or "true 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[47].

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 shall 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 fractures.

NOTE At present, there appears to be insufficient quantitative data in the literature demonstrating the influence of bending upon creep and stress rupture life. Any organization with such information is encouraged to forward it to ISO/TC 164[43].

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

6.2 Extension and elongation measuring devices.

6.2.1 Extension measuring device.

In uninterrupted tests, the extension 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 be non-contacting (e.g. a non-contacting optical or laser extensometer).

It is recommended that the extensometer be 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 duration 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 capable of measuring extension of one side of the test piece or, preferably, on opposite sides of the test piece.

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

When the extension 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 extension has occurred completely within the reference length of the test piece. Percentage creep extension 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.

Care should be taken to avoid spurious negative creep when using nickel base alloy extensometers. See the Code of Practice[42].

For low creep strain measurements, e.g. ≤1 % strain, on test pieces with short gauge lengths, careful consideration should be given to ensure that the measuring device used has sufficient resolution and accuracy over the range of use.

NOTE 1 Information on the long-term stability of transducers used for creep testing and accreditation issues is given in References [40] and [41].

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

6.2.2 Elongation measuring device.

In interrupted tests, periodically unload the test piece, 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 ΔL_r or 0,01 mm, whichever is the greater. After this measurement, the test piece may be first reheated and then reloaded.

6.3 Heating device, temperature measuring equipment and calibration.

6.3.1 Permissible temperature deviations.

The heating device shall heat the test piece to the specified temperature, T. The permitted deviations between the corrected measured temperature, T_c, 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_c and T and maximum permissible temperature variation along the test piece

Specified temperature T °C	Permitted deviation between T_c and T °C	Maximum permissible temperature variation along the test piece °C
$T \leq 600$	±3	3
$600 < T \leq 800$	±4	4
$800 < T \leq 1\,000$	±5	5
$1\,000 < T \leq 1\,100$	±6	6

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

The corrected measured temperatures, T_c, are the temperatures measured at the surface of the parallel length of the test piece, errors from all sources, including drift (see Annex A), being taken into account and any systematic errors having been corrected.

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 on the test piece instead of measuring the temperature at the surface of each individual test piece.

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 ±3 °C.

In the interrupted test, the variation of the room temperature during all measurements of the gauge length should not exceed ±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. The temperature measuring equipment shall have an accuracy equal to or better than ±1 °C.

For thermocouples, in the absence of measuring instruments with cold junction compensation, cold junction temperatures, normally at 0°C, shall be measured to within 0,5 °C.

Many laboratories maintain the cold junction above ambient temperature. Whatever its temperature, it shall remain stable and appropriate compensation shall be applied to determine the temperature measured by the thermocouple.

NOTE Information concerning drift of thermocouples is given in Annex A and methods of calibration of thermocouples are given in Annex B.

For indirect methods of temperature measurement e.g. pyrometry, thermal cameras or resistivity techniques, it shall be demonstrated that traceability is provided to the SI System of temperature measurement and that the above criteria for accuracy and resolution can be achieved.

6.3.2.2 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.

6.3.2.3 Single test piece machines.

In single test piece machines, with thermocouples used for temperature measurement, at least two thermocouples should be used for test pieces with a parallel length less than or equal to 50 mm. 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.

6.3.2.4 Multiple test piece machines.

In multiple test piece machines, with thermocouples used for temperature measurement, at least one thermocouple should 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 ±2 °C up to 800 °C and ±3 °C above 800 °C.

6.3.2.5 Notched test pieces.

Temperature measurement of notched test pieces shall be in accordance with either 6.3.2.3 or 6.3.2.4. It is recommended that one thermocouple be placed close to the notch.

NOTE Details about testing notched test pieces are given in Annex C.

6.3.3 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.

Precautions shall be taken to minimize contamination and physical damage of thermocouples. Insulators and/or insulation shall be maintained in a clean state to also minimise contamination and prevent conduction.

Information concerning different types of thermocouples is given in IEC 60584-1[7].

The use of rare metal thermocouples, preferentially of type S or R, is recommended for temperatures equal to or greater than 400 °C[44].

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 without cutting back exposed wire and re-calibrating.

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 use of type N base metal thermocouples is not permitted at temperatures above 760 °C[44].

Where the drift exceeds the following values within the calibration period, either more frequent calibrations should be carried out or a correction be applied to the temperature indicated by the thermocouple. See Annex A and References [48] to [52].

— ±1 °C for $T \leq 600$ °C;

— ±1,5 °C for 600 °C $< T \leq 800$ °C;

— ±2 °C for 800 °C $< T \leq 1\,100$ °C.

Records of drift shall be recorded and available on request.

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

NOTE 2 Reference can be made to ASTM E633[8].

NOTE 3 This clause is not applicable in the case of indirect temperature measurement.

NOTE 4 Other types of thermocouples are available but their suitability will be demonstrated.

6.3.4 Calibration of the thermocouples.

NOTE 1 Further information relating to thermocouples calibration is given in Annex B.

The calibration period for rare metal thermocouples in repeated use for short duration tests (typically 500 h or less) shall not exceed 13 months. Otherwise the period for calibration shall be as follows:

— 4 years for $T \leq 600$ °C;

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

— 1 year for $T > 800$ °C.

The re-use of base metal thermocouples is not permitted without re-calibration after each test unless experience has shown that errors due to drift do not exceed the limits in 6.3.3 for the test conditions in question, in which case they may be calibrated at intervals not exceeding 500 h use.

If the test duration exceeds the above calibration periods, the thermocouple shall be calibrated upon completion of the test.

It shall be demonstrated that the error of the thermocouple used has been established at the test temperature or is typical for a range containing the test temperature. Thermocouples showing errors in excess of 1 °C may be used provided the appropriate corrections are made.

If a thermocouple is rewelded, the thermocouple shall be recalibrated before use.

NOTE 2 Base metal thermocouples can be cut back removing any exposed wire and will be re-calibrated before use.

7 Test pieces

7.1 Shape and dimensions

7.1.1 Shape and dimension of smooth test pieces

Normally test pieces with circular cross-sections shall be used; see Figure 2.

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

In special cases, the cross-section of the test piece may be square, rectangular or of some other shape. For these specific test pieces, the provisions specified for cylindrical test pieces with a circular cross section shall be applied.

In general, L_r 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 mm^2.

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

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

— 0,005*b* 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_0) 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 %.

7.1.2 Shape and dimension of notched test pieces

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

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* ≤ 6	0,02
6 < *D* ≤ 10	0,03
10 < *D* ≤ 18	0,04
18 < *D* ≤ 30	0,05

[a] Maximum deviation between the measurements of a transverse dimension determined along the entire parallel length of the test piece[3].

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

Dimensions in millimetres

Nominal dimension *b*	Shape tolerances[a]
3 < *b* ≤ 6	0,02
6 < *b* ≤ 10	0,03
10 < *b* ≤ 18	0,04
18 < *b* ≤ 30	0,05

[a] Maximum deviation between the measurements of a transverse dimension determined along the entire parallel length of the test piece[3].

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.

NOTE To avoid the fracture position too near to the end of gauge length, it can be sensible to exploit half the shape tolerance for a tapering of the test piece towards the centre of the gauge length.

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

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 ±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 force 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 ±1 %.

NOTE In some cases, it can 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 test piece with small collars is used [see Figures 2 c) and d)].

7.5 Determination of the reference length, L_r

Within this document, the various percentage extension/percentage elongation values (see 3.11 to 3.22) are expressed as percentages of the reference length, L_r. The value of the reference length depends on the test piece geometry and the type of extensometer that is used. Two different cases need to be distinguished.

Case 1 — Original gauge length, L_o, and/or extensometer gauge length, L_e, inside the parallel length, L_c.

This case is illustrated in Figure 2 a) and b), where the diameter of the test piece is constant within the original gauge length and the extensometer gauge length (e.g. due to a respective positioning of original gauge length marks or an extensometer that is attached only to the parallel length of the test piece). The reference length, L_r, for calculation of percentage elongations is then given by Formula (9):

$$L_r = L_o \tag{9}$$

Similarly, the reference length, L_r, for calculation of percentage extensions is expressed by Formula (10):

$$L_r = L_e \tag{10}$$

Case 2 — Original gauge length, L_o, and/or extensometer gauge length, L_e, outside the parallel length, L_c.

This case is illustrated in Figure 2 c) and d), where test piece sections with diameter variations (like shoulders of the test piece or ridges for extensometer application) form part of the original gauge length and the extensometer gauge length. The reference length, L_r, for calculation of percentage extensions/elongations should be calculated using Formula (11) to consider the strain contributions of the shoulders/ridges (see Figure 2 d):

$$L_r = L_c + 2\sum_i \left[\left(\frac{D}{d_i} \right)^{2n} l_i \right] \tag{11}$$

where

n is the stress exponent at the test temperature for the material under investigation (if this is not known, use $n = 5$);

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);

d_i is the test piece diameter in the central cross section of the respective length increment, l_i.

In the case of e.g. rectangular or hollow cross section, Formula (12) should be used:

$$L_r = L_c + 2\sum_i \left[\left(\frac{S_o}{S_i} \right)^n l_i \right] \quad (12)$$

where

S_o is the original cross-sectional area of the parallel length;

S_i is the test piece cross-sectional area of the respective length increment l_i.

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. On the contrary, a recalculation becomes necessary when relevant changes of the stress exponent, n, occur either due to testing of a different material or due to testing in an extended range of initial stresses and test temperatures.

NOTE If the difference between the calculated reference length and the extensometer gauge length, L_e, or the original gauge length, L_o, is less than 0,5 % then L_r can be chosen as equal to L_o or L_e .

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 extensometer shall be at thermal equilibrium.

This condition shall be maintained for at least 1 h 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.

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 ±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 force or at room temperature.

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

8.3 Test interruptions

8.3.1 Planned interruptions of the test

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

NOTE An example is given in 8.4.2.

8.3.2 Multiple test piece machine with several test pieces in line

After a test piece has fractured, 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 Combined test

The test shall be performed initially with continuous strain measurement until the completion of primary creep. If desired, the test piece may then be moved to another machine for continuation as an interrupted test. See also 8.4.2 and Annex E[45][46].

8.3.4 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.

NOTE The initial applied force can be maintained during these interruptions to minimize disturbance to the extensometry. If necessary the load can be reduced during a power interruption and the test re-loaded when power is resumed and the test temperature achieved. It is prudent to maintain a force of at least 10 % to ensure the loading string remains aligned.

8.4 Recording of temperature and elongation or extension

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 and extension

Either a continuous record of extension 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 percentage creep extension 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 extension, e_i, shall be determined.

For the determination of the initial plastic extension, the elastic extension has to be subtracted. The elastic extension should be determined from a stepwise measurement procedure during loading, or it can be taken over from a tensile test at elevated temperature in accordance with ISO 6892-2. The elastic extension can alternatively be determined from a partial unloading procedure immediately after loading if the plastic extension is lower than 1 %.

To determine the percentage initial plastic extension, e_i, in the case of the interrupted creep test, a tensile test at elevated temperature in accordance with ISO 6892-2 and ISO 6892-1 shall additionally be performed at each creep test temperature with the exception from ISO 6892-2 that the strain rate should be similar to the loading rate used 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, 500 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 or extension-time diagram

On the basis of records of time and elongation or extension, an elongation-time diagram or extension-time diagram, respectively, 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 percentage elongation after fracture shall be considered invalid if the test piece fractures 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

The resulting values shall be expressed in accordance with the following requirements concerning the rounding rules:

— specified temperature (T): to 1 °C;

— diameter (D): to 0,01 mm;

— ratio (L_r/D): to one decimal place;

— reference length (L_r): to 0,1 mm;

— initial stress (R_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 extension (e_e, e_i, e_f, e_{fu}, e_k, e_p, e_{per}, e_{pu}): 3 significant figures;

— percentage permanent elongation (A_{per}): 3 significant figures;

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

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

11.2 Final uncertainty

Because the uncertainty of measurement 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 document, i.e. ISO 204:2018;

— 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 corrected measured temperature, if it is outside the permitted limits;

— initial applied stress;

— constant applied force or constant applied stress;

— test results;

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

— percentage initial plastic elongation or extension;

— 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 (single test piece machine, multiple test piece machine, etc.);

— force application time;

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

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

— percentage elastic and anelastic elongation or extension 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;

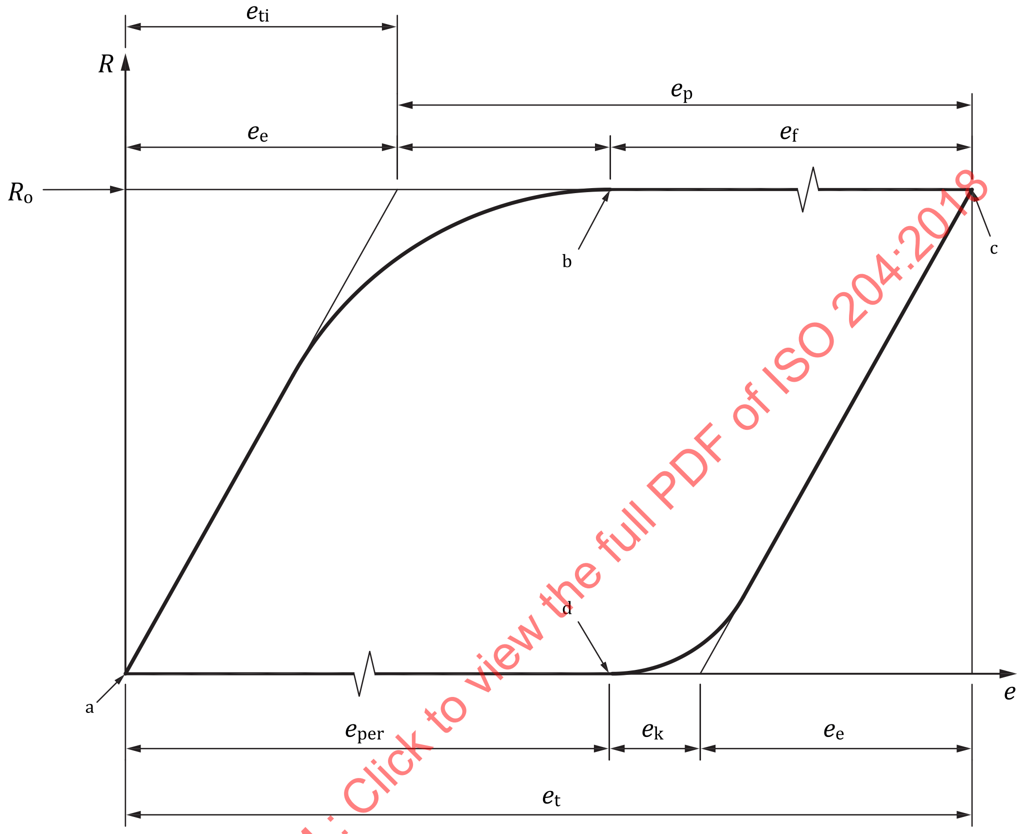
— type of extensometer;

— 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 document shall not be adjusted to take account of uncertainties of measurement, unless specifically instructed otherwise by the customer (see Annex D).

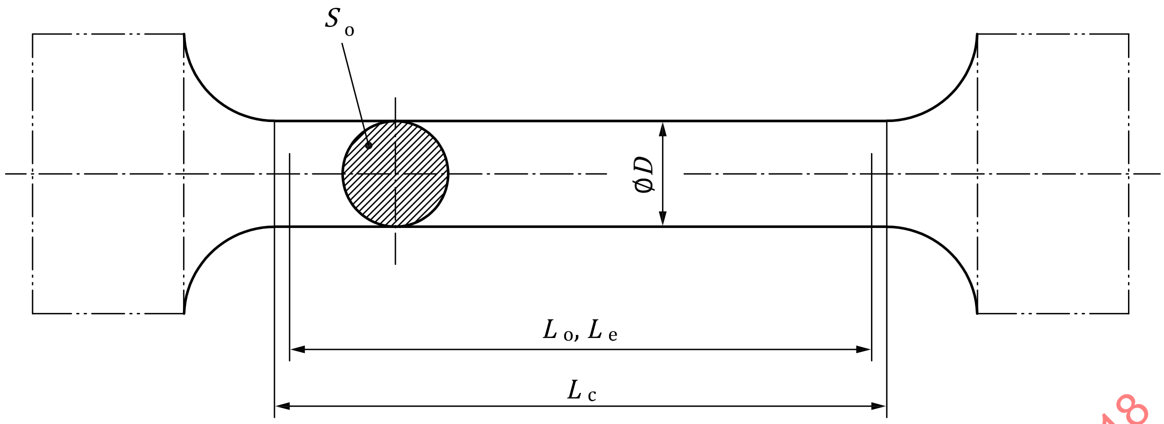
12.5 The estimated measurement uncertainties shall not be combined with test results to assess compliance with product specifications, unless specifically instructed otherwise by the customer (see Annex D).



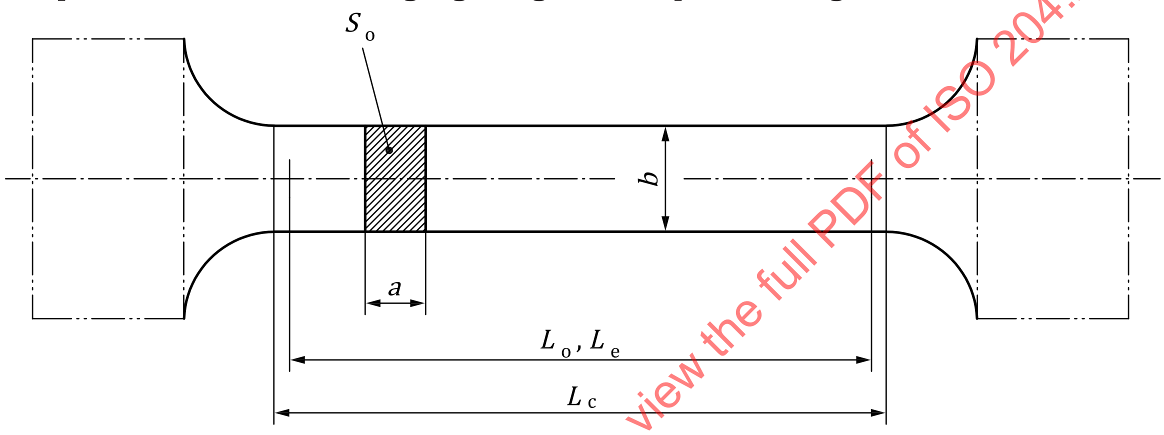
Key

R	stress
R_o	initial stress
e	percentage extension
e_t	percentage total extension
e_e	percentage elastic extension (after loading with initial stress)
e_p	percentage plastic extension
e_f	percentage creep extension
e_{ti}	percentage initial total extension
e_i	percentage initial plastic extension
e_k	percentage anelastic extension
e_{per}	percentage permanent extension
a	Start of loading.
b	End of loading.
c	Start of unloading.
d	End of unloading.

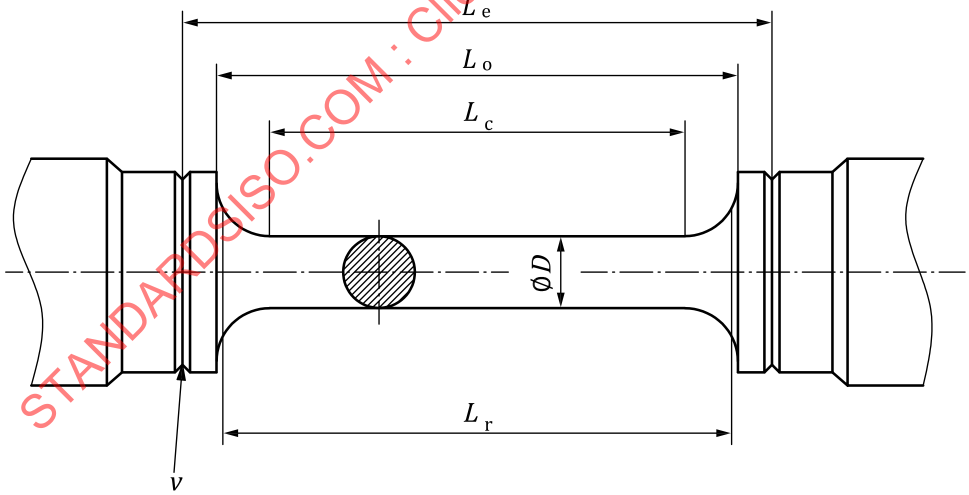
Figure 1 — Schematic stress — Extension diagram



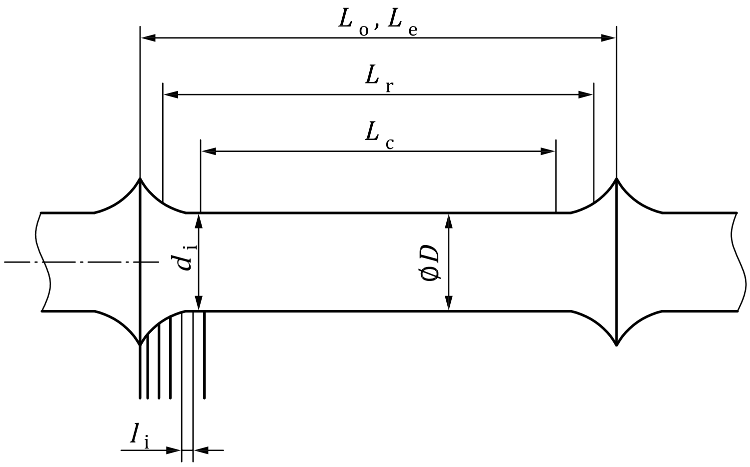
a) Test piece with shoulders and gauge length inside parallel length and round cross section



b) Test piece with shoulders and gauge length inside parallel length and rectangular cross section



c) Test piece with shoulders and gauge length outside parallel length



d) Test piece with collars

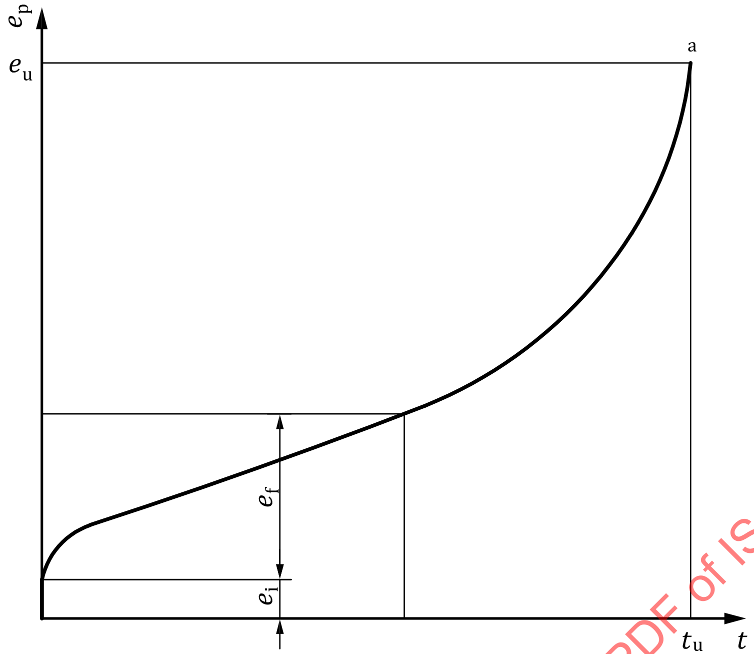
Key

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

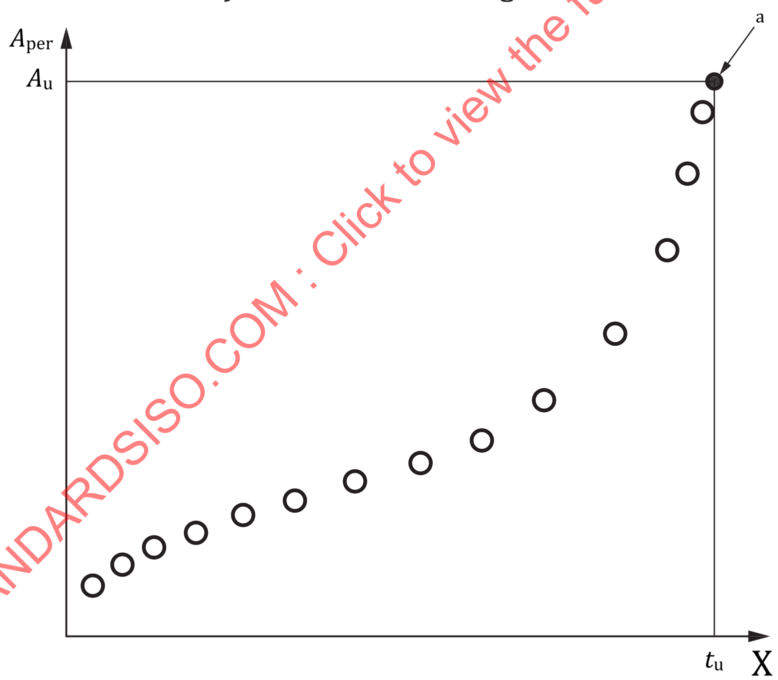
NOTE 1 L_r is determined in accordance with Formula (11) or Formula (12), as appropriate.

NOTE 2 The shape of the grip ends is given for information only.

Figure 2 — Examples of test pieces



a) Extension-time diagram



b) Elongation-time diagram

[a] Fracture.

Figure 3 — Creep curves

Annex A
(informative)

Information concerning drift of thermocouples

A.1 General

The electromotive force (emf) produced by a thermocouple can change with long term exposure at high temperature; this behaviour is colloquially known as “drift”.

If the drift characteristics of the particular type of thermocouple at a specified creep test temperature are known then that information may be used to adjust furnace temperature on a creep testing machine as time elapses to ensure that the test remains within the limits given in Table 2. It should be noted that there are several contributions to the uncertainty budget, of which drift may be one of the larger components.

A.2 Consequences of drift

Until about twenty years ago, many creep laboratories **controlled** the temperature of creep furnaces using platinum resistances thermometers (PRTs) whilst **monitoring** the temperature of the test piece with thermocouples. In general, PRTs are not prone to drift and thus the creep test temperature remained stable within the specified tolerances given in the testing standards.

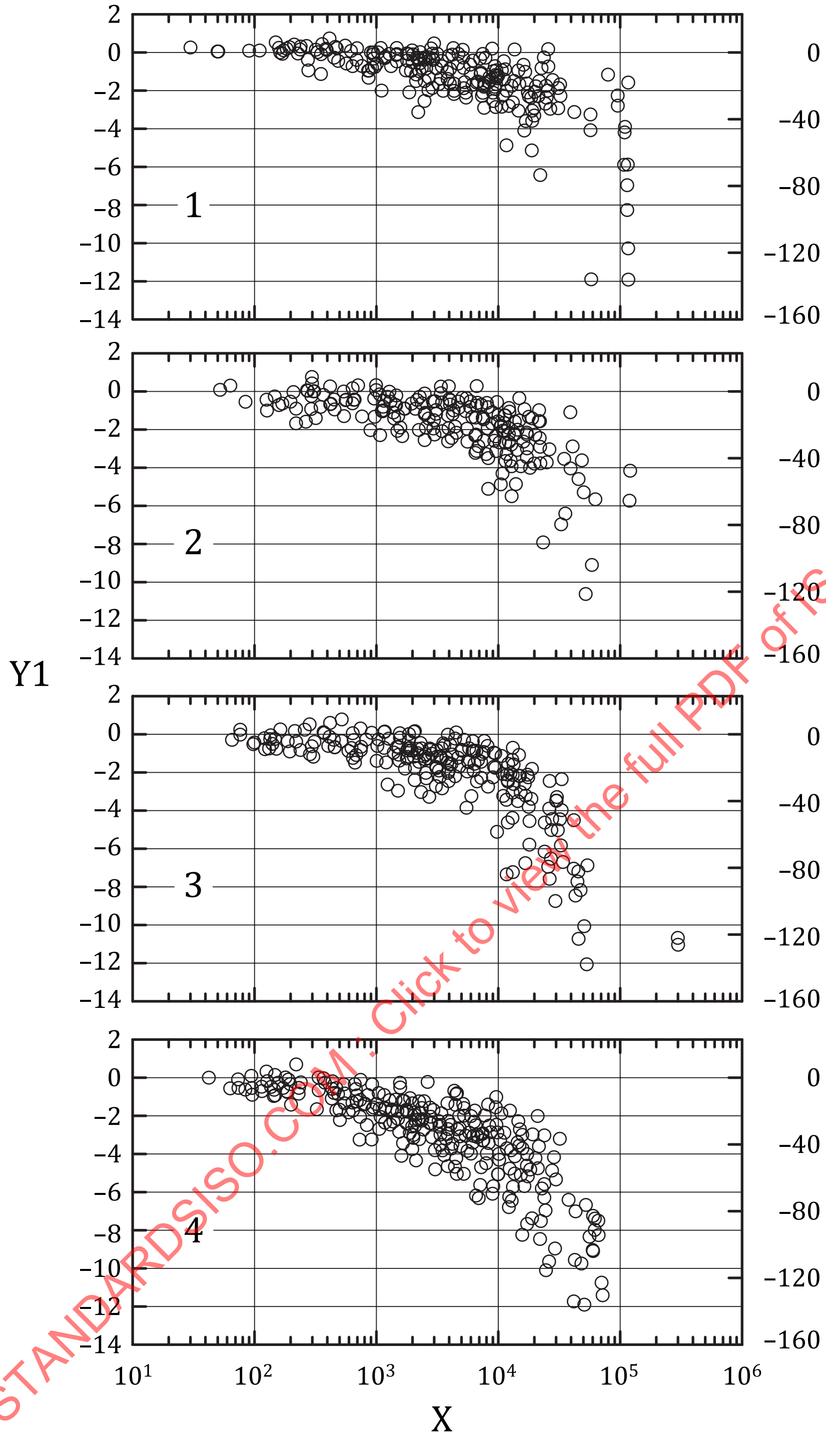
In most creep tests undertaken today, the furnace temperature is **controlled** by a thermocouple attached to the test piece. Thus if the emf of the control thermocouple drops off due to drift, the electronics of the temperature controller senses the reduction in the emf and automatically increases the power to the furnace so as to restore the indicated emf to the set point value. As a consequence, if the thermocouple output drifts downwards, the true temperature of the creep test actually systematically increases. Depending upon the magnitude of the drift, the test may deviate outside the test tolerances specified in the testing standard.

The consequence of the test temperature continually increasing is that the measured creep life will be shorter and the creep rate higher than in a test carried out at a constant temperature; thus, in general, creep data is conservative and such data used for design of safety critical components will err on the side of safety.

A.3 Drift data

During the 1960-70s, creep laboratories started to realize that thermocouple drift could result in tests not complying with the temperature tolerances specified in the standards and thus it became common practice to recalibrate intact thermocouples after a creep test was completed. In Europe, the use of type K (Chromel/Alumel) thermocouples was largely discouraged for long term creep testing during the 1960s because it was recognised that they were prone to significant drift[55][60].

Post creep test calibration data was reported in the UK for type R thermocouples[55] over a range of creep test temperatures from 400 °C to 850 °C. Although the data shows a large amount of scatter, a downward drift trend was indicated with approximately a drift of −3 °C ± 2 °C at 600 °C. In addition, in Japan, similar data was recorded for type R & S thermocouples.[61][64][65][69] The Japanese NRIM/NIMS Creep Data sheet (CDS) project is still ongoing and an update of the changes in thermal electromotive force of type PR thermocouples (Pt–Pt 12,8 %Rh) and type R after service for creep between 500 °C to 900 °C was presented at the ECCC conference in 2005[64]. An example graph is shown in Figure A.1, and it can be seen that several results are now available for lives exceeding 100 000 h. Drift values of up to 12 °C were reported.



Key

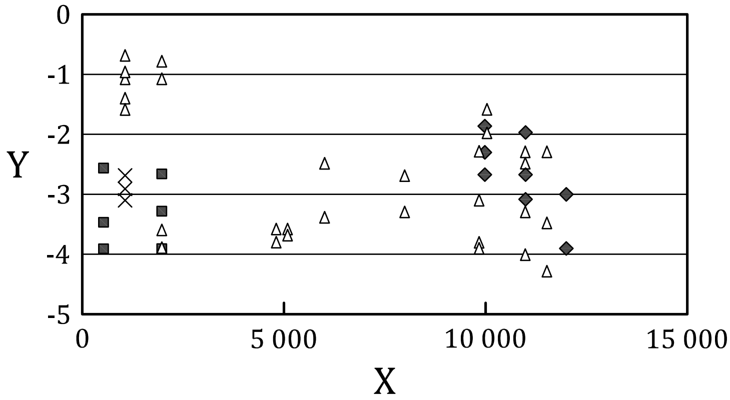
X duration of creep test, h

Y1 calibration drift, °C

Y2 calibration drift, µV

Figure A.1 — Type PR thermocouple drift data measured after creep testing (*Miyazaki, H and Kimura, K, 2005*)[64]

In addition, some limited data has been published for drift data after recalibration of type R thermocouples following creep test at NPL[63]. See Figure A.2.



Key

- ◆ 750 °C
- ■ 800 °C
- △ 850 °C
- × 900 °C
- X duration of creep test, h
- Y calibration drift, °C

Figure A.2 — Type R thermocouple drift data measured after creep testing[63]

Type N thermocouples are now being widely used for plant monitoring and creep testing, but as yet no systematic drift data has been published in the temperature range used for creep testing. To address this deficiency, the High Temperature Mechanical Testing Committee instigated some drift measurements on sheathed type N thermocouples, initially at 650 °C at EDF, Gloucestershire and at 750 °C at NPL.

Preliminary results from the isothermal measurements undertaken at EDF Energy which have been running for about 25 000 h indicate that drift of ~2 °C can be encountered which is not significantly less than that measured in type K thermocouples.

Information on the performance of thermocouples used in creep testing is also given in other sources[57][58][59][67][68].

A.4 Concluding remarks

In general, the higher the creep test temperature, the greater the drift and it is clear that, unless an allowance is made for drift, many creep tests will not comply with the tolerances specified in Table 2.

A number of laboratories have measured drift data after creep testing but have not published the information. It would be helpful if such information were to be made publicly available.

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 force (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[7] reference table for that temperature. Both strategies involve 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 60584-1:2013[7], 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 of dimensions of notched test pieces with circular cross-sections and with an elastic stress concentration factor K_t = 4,5 ± 0,5[10]

Dimensions in millimetres

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

For dimensions deviating from Table C.1, the test piece 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 ± 12,5 %.

Additional information about stress concentration factors can be found in References [11] and [12].

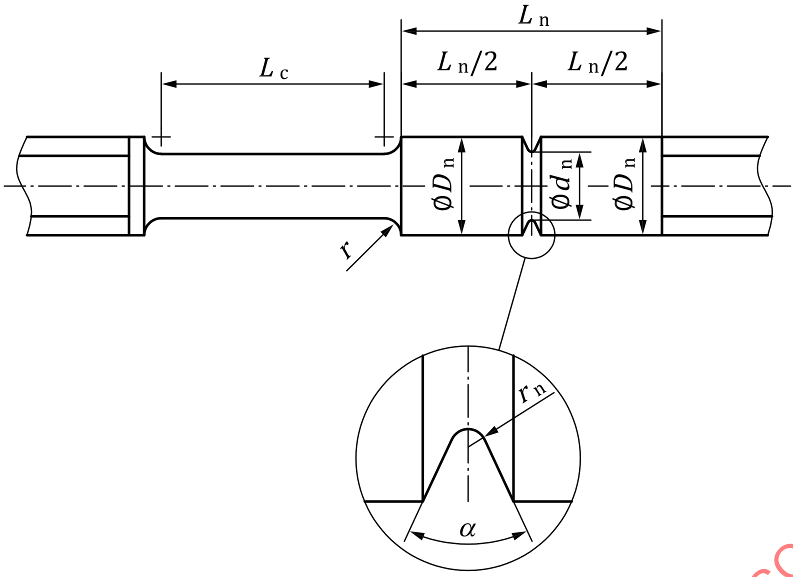
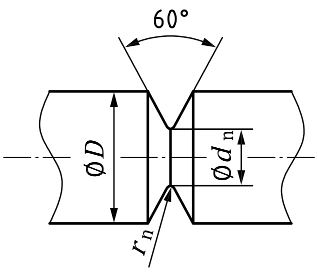


Figure C.1 — Combined notched and unnotched test piece

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 considered that the notch geometry, type E, shown on Figure C.2, is suitable for assessing whether a material notch strengthens or weakens[13].



Type	DIN	BS	E
D/d_n	1,25	1,41	$\sqrt{1{,}25\times1{,}41}=1{,}33$
D_n/r_n	50	35	$\sqrt{50\times35}=42$

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

The elastic stress concentration factor[10] is calculated using Formula (C.1):

$$K_t = 1 + \left[\frac{1}{2} \times \frac{\frac{r_n}{d_n}}{\frac{D_n}{d_n} - 1} + 2 \times \frac{r_n}{d_n} \times \left(1 + 2 \times \frac{r_n}{d_n} \right)^2 \right]^{-\frac{1}{2}} \quad \text{(C.1)}$$

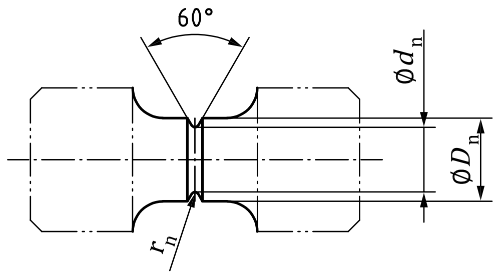


Figure C.3 — Schematic diagram of a notched test piece with a circular cross-section

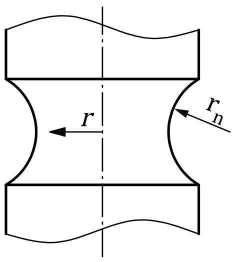
C.3 Blunt circumferential notches

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[14]. A *Code of Testing Practice for Notched Bar Creep Rupture Testing* was produced by a Working Group of the High Temperature Mechanical Testing Committee (HTMTC) in the early 1990s[15]. This latter document was subsequently revised[16], based on an EU-funded project[17].

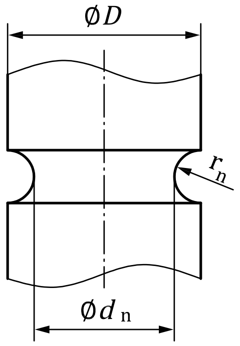
The Code of Practice has additionally been updated to cover creep strain measurement, which may be undertaken using axial, or diametral extensometers[18]. Further information has also been published relating to diametral strain measurement on notched creep test pieces[19][20] and calibration of diametral extensometers[21].

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.4.

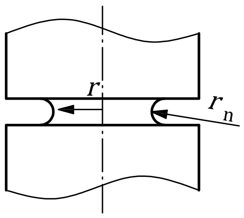
The interpretation of the data generated using such notches is complex and is discussed in detail by Webster, *et al.*[18].



a) Blunt



b) Semi-circular



c) Parallel-sided

Figure C.4 — Three possible types of Bridgman notch[18]

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 Purpose

This annex gives guidance on how to estimate the uncertainty of the measurements undertaken in accordance with this document using a material with known creep properties. 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, BCR425[32], to assess conformance with this document.

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 ISO/IEC 17025[6]).

In addition, two important documents have emerged from ISO Standards Committees, i.e. the ISO 5725 series [5] and the *Guide to the expression of uncertainty in measurement*. Such documents largely use the terms and vocabulary given in VIM, 1993[2][1)].

In 1995, the *Guide to the expression of uncertainty in measurement* (hereafter, “GUM”) was published jointly by several authoritative standards bodies, namely BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML. In 2008, the GUM was reissued with minor corrections as ISO/IEC Guide 98-3[1]. It 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 number of organizations to produce

1) 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)*.

simplified versions of the GUM, e.g. the National Institute of Standards and Technology (NIST) in the USA[22], the United Kingdom Accreditation Service (UKAS) in the UK[23] and the British Measurement and Testing Association (BMTA[24]), 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*[25] and *Estimating Uncertainties in Testing* [26]. The approach adopted here for the Tensile Uncertainty Budget[27] is similar to that proposed for a creep testing uncertainty budget used in association with the Creep Certified Reference Material, CRM BCR425[28],[31]. Comprehensive statements of uncertainty have also now been published as part of the EU-funded project *Uncert* [29] and an additional document was issued covering creep uncertainty as a CEN-endorsed Technical Workshop Agreement, CWA 15261-3[9], which will shortly be available as ISO TR 15264[9][2].

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.

2) Under preparation.

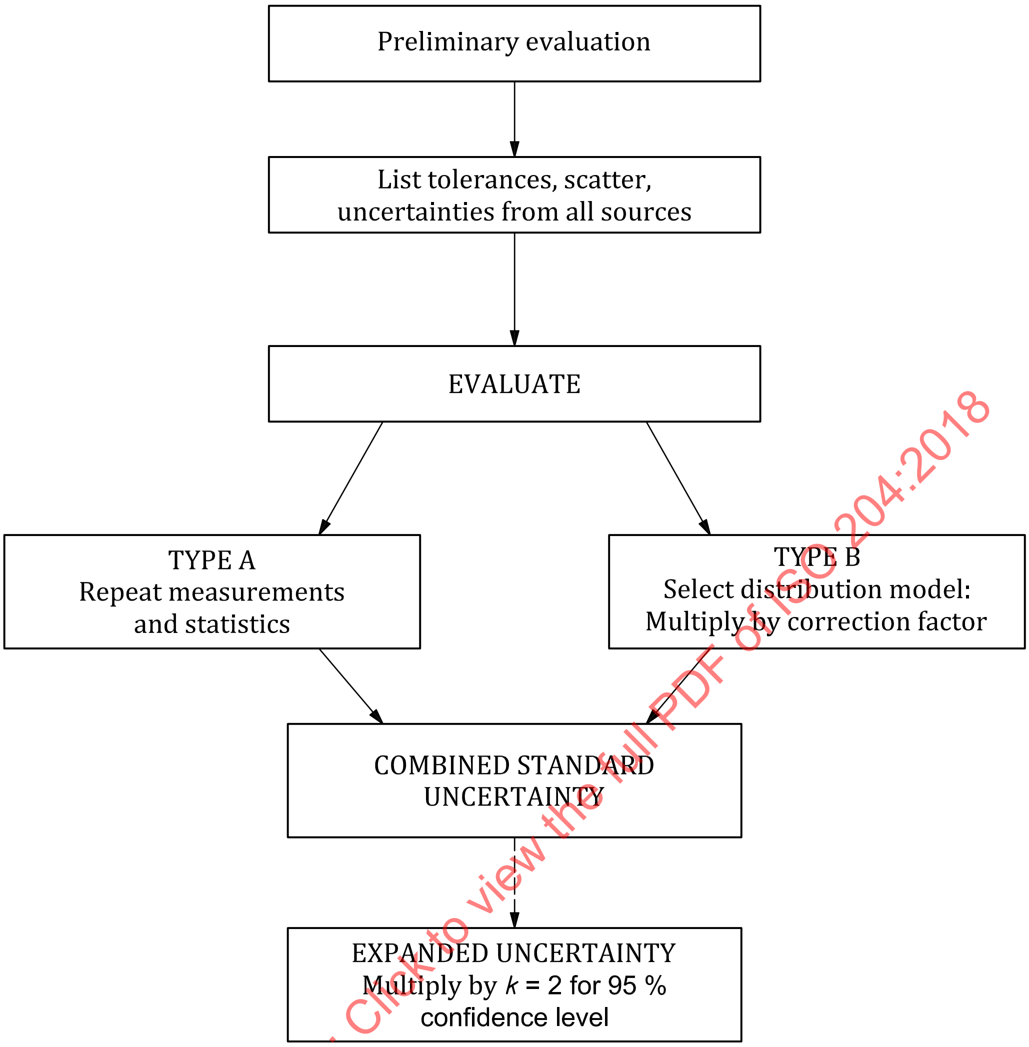


Figure D.1 — Outline procedure for estimation of uncertainty

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.

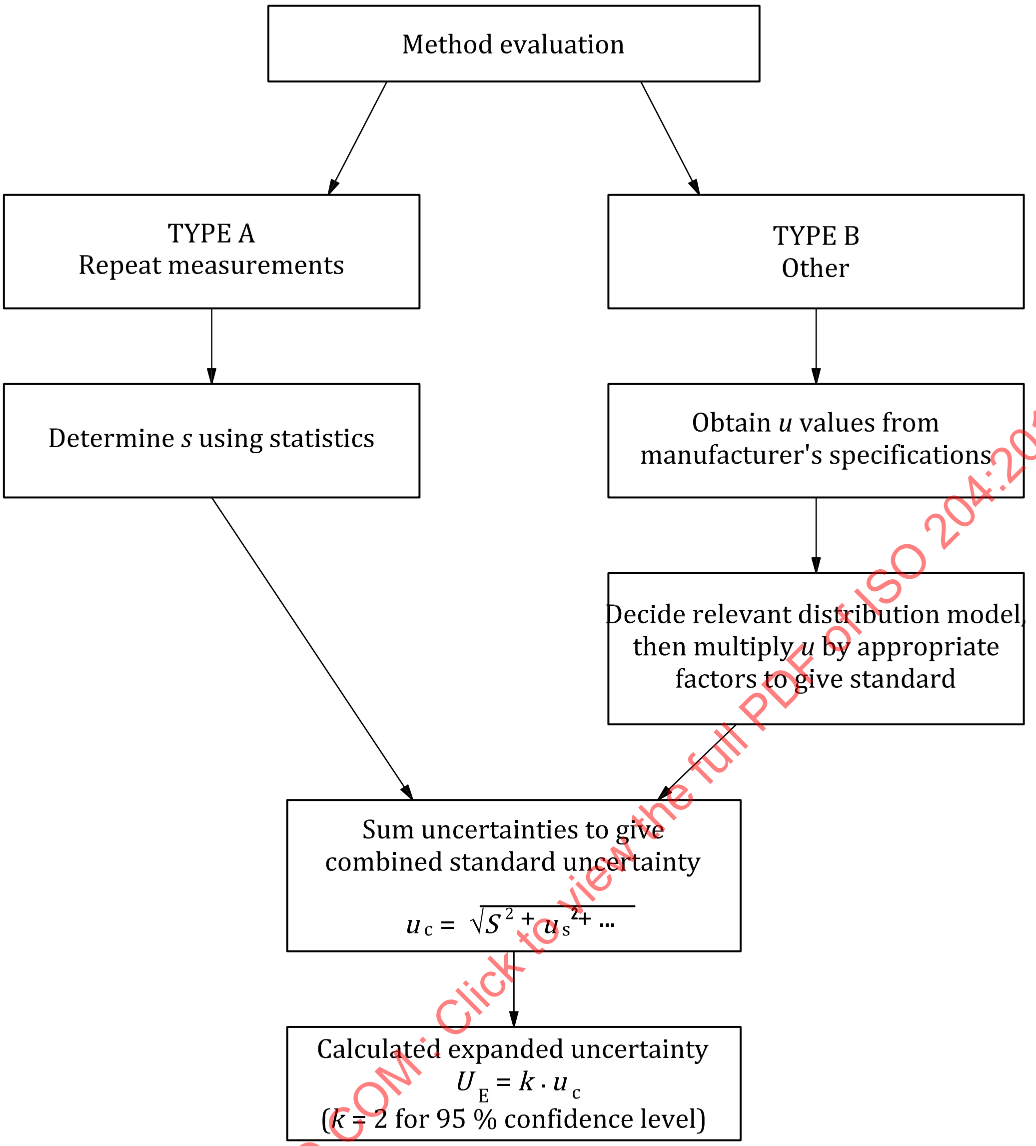


Figure D.2 — Detailed procedure for estimating uncertainty in accordance with the GUM

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, σ, and the temperature, T, by Formula (D.1):

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

where

A is a material constant;

n is the stress index in the Norton creep law;

Q is the creep activation energy;

R is the universal gas constant.

NOTE For the purpose of Annex D, the above symbols are used and do not have the same definitions as those listed in Table 1.

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 σ and T in the two separate components of Formula (D.1). Tolerances for σ 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 this document.

Using Formula (D.1), it has been shown elsewhere[28] that for the solid solution nickel base alloy, Nimonic 75 (CRM BCR425), 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 document, the expanded measurement uncertainty U_E = 20,2 % at the 95 % confidence level.

Similarly, Granacher and Holdsworth[30] 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 h. 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 is 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

In addition, other factors 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. 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 conformance with this document.

D.4 A reference material for creep testing

D.4.1 General

During recent years, the benefits of the use of Certified Reference Materials (CRM) 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[31]; see Table D.2.

Table D.2 — Certified values for the Nimonic 75 Creep Reference Material, BCR425

Property[a]	Certified value[b]	Uncertainty[c]
Creep rate at 400 h	$71{,}8 \times 10^{-6}$ h^{-1}	5×10^{-6} h^{-1}
t_{p2}	278 h	16 h
t_{p4}	557 h	30 h

[a] Testing conditions: T = 600 °C, σ_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 BCR425 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 BCR425, a test undertaken in accordance with this document 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 ΔT = ±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 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 [33], [34] and [35]).

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	Data range	Average	Total uncertainty
Time to rupture (h)	238,6 ~ 460,8	333,9	±59
Elongation (%)	6,3 ~ 13,4	10,3	±5,2
Reduction of area (%)	24,7 ~ 38,9	33,7	±8,2

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

Annex E
(informative)

Representation of results and extrapolation

E.1 General

This annex summarizes important information which should help the user to choose a suitable test type for their needs (i.e. uninterrupted, interrupted or combined) and apply the established methodology developed within the European Creep Collaborative Committee[36].

E.2 Symbols for strength values and their calculation

E.2.1 Strain

In most cases, the anelastic strain, e_k, is negligible and there is no difference between the plastic strain, e_p, and the permanent strain, e_{per}.

E.2.2 Creep rupture strength

The creep rupture strength at a specified test temperature, T, is the applied stress, R_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, R_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 force.

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 Creep testing in single test piece machines and/or multiple test piece machines

Creep tests on smooth (unnotched) test pieces may be performed in **uninterrupted** mode[44], typically in a single creep test piece machine with continuous extension measurement, or in **interrupted** mode[44] typically in multiple creep test piece machine.

In the interrupted mode for elongation measurement, the test piece is removed from testing machine and cooled down. Subsequently, the test piece is re-mounted, heated and loaded again.

Alternatively, creep tests may be performed in multiple test piece machine with continuous extension measurement for each test piece.

To obtain sufficient information about primary creep, test pieces may be tested in a single test piece machine with continuous extension measurement and high resolution up to a certain strain and then be relocated into a multiple test piece machine to test up to rupture in interrupted mode.

Primary creep information is necessary due to the fact that local stresses in notches cause stress redistribution due to plastic deformation. At high temperature superimposed creep deformation contribute to stress redistribution. To recalculate stress redistribution in components, creep equations are recommended which consider the primary and secondary creep regime.

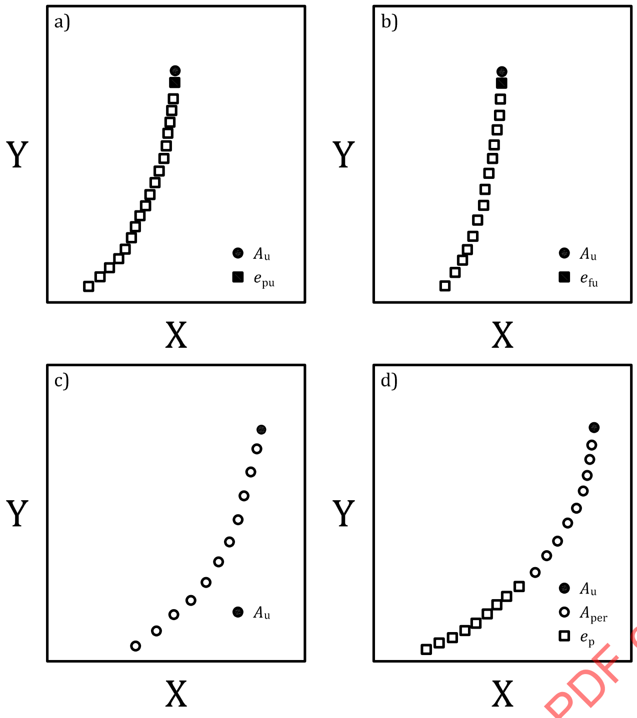
The experimental results of creep tests in uninterrupted mode are typically displayed in a logarithmic percentage plastic extension-time diagram [Figure E.1 a)] and/or a logarithmic percentage creep extension-time diagram [Figure E.1 b)].

Experimental results of creep tests in interrupted mode are typically displayed in a logarithmic percentage permanent elongation-time diagram [Figure E.1 c)].

The experimental results of combined testing techniques are displayed according to [Figure E.1 d)].

The percentage plastic extension at creep rupture time, e_{pu}, may be determined from the continuous measurement at uninterrupted creep tests of the increase of length at creep rupture time, t_u. A corresponding value, e_{fu}, may be determined from percentage creep extension at creep rupture time.

NOTE The test piece will not be moved from a single creep test piece machine to a multiple creep test piece machine until it has been assessed that primary creep has been completed.



Key

X log t

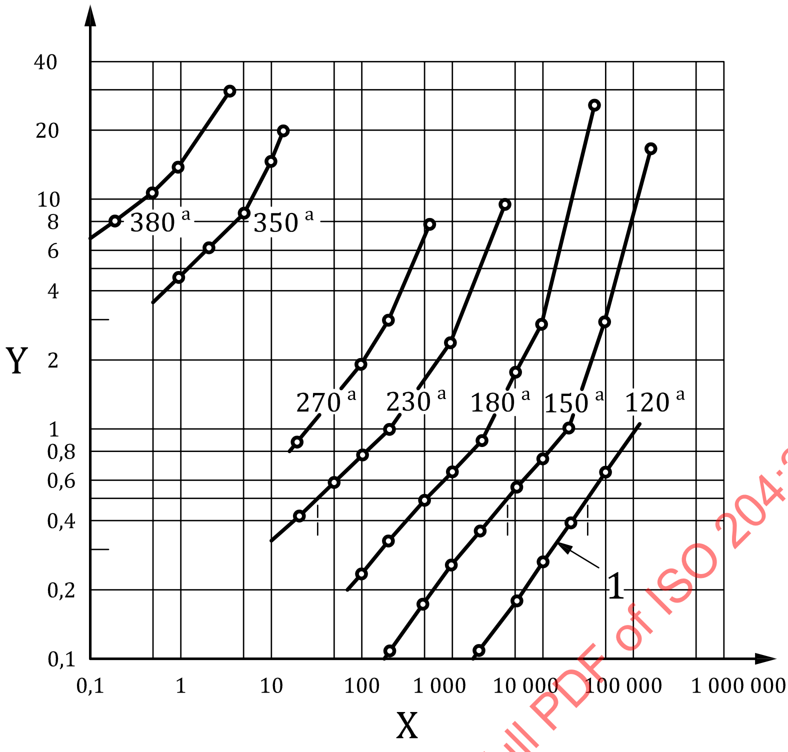
Y log e_p

Figure E.1 — Schematic representation of creep curves generated in non-interrupted creep test mode (a, b), interrupted creep test (c) and creep test to be started in non-interrupted creep test mode and continued as interrupted creep test (d)

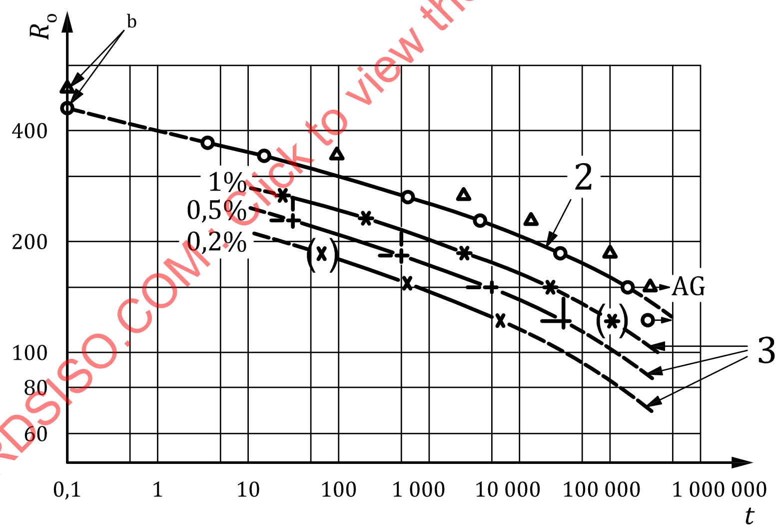
E.4 Evaluation

E.4.1 General

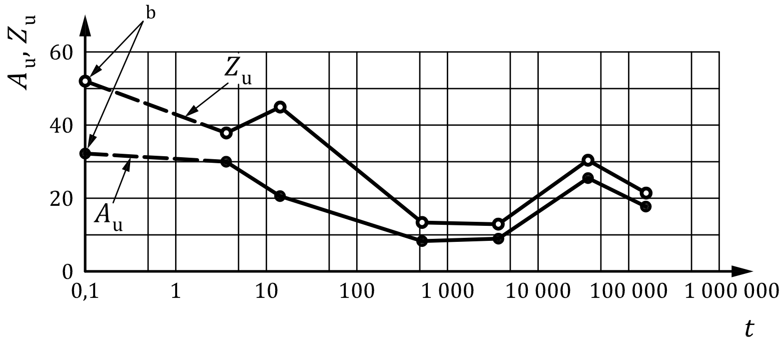
The experimental results of an individual material for one test temperature can be displayed and evaluated in a number of diagrams (see Figures E.2 and E.3). In these diagrams, extrapolated curves should be dashed while extrapolated points should be in parentheses. In E.5, some remarks about the extrapolation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



a) Creep diagram



b) Creep rupture and stress-to-specific-plastic-strain diagram



c) Creep rupture elongation diagram

Key

1	creep curve	o→AG	test stopped without rupture[c]
2	creep rupture curve	o→	test running
3	stress-to-specific-plastic-strain curve	△→AG	test stopped without rupture
o	smooth test pieces (rupture)	△→	test running
△	notched test pieces (rupture)	– –	extrapolated

Points in ( ) are based on extrapolated data

Horizontal axis: t, time (hours); E.2 c): t_u (hours)

[a] Initial stress in MPa.

[b] Data from tensile test at elevated temperature.

[c] AG from the German "*ausgebaut*" — test stopped without rupture.

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

E.4.2 Logarithmic creep diagram

In order to display creep curves, the percentage plastic extension, e_p, or the percentage permanent elongation, A_{per}, may 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. $t_{p0,2}$, are plotted in dependence of initial stress, R_o, in logarithmic scales [see Figure E.2 b)]. The curve should be smooth. From this diagram, the stress-to-strain, $R_{px,t,T}$, is taken.

To determine the creep rupture diagram, the rupture time, t_u, is plotted in dependence of initial stress, R_o, 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, R_o, of notched test pieces can be plotted as a hint in this diagram. Additional judgments of the material behaviour can be achieved in this way.