



BSI Standards Publication

Superconductivity

Part 19: Mechanical properties
measurement — Room temperature
tensile test of reacted Nb_3Sn
composite superconductors

National foreword

This British Standard is the UK implementation of EN 61788-19:2014. It is identical to IEC 61788-19:2013.

The UK participation in its preparation was entrusted to Technical Committee L/-/90, Super Conductivity.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Date	Text affected
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English version

**Superconductivity -
Part 19: Mechanical properties measurement -
Room temperature tensile test of reacted Nb₃Sn composite
superconductors
(IEC 61788-19:2013)**

Supraconductivité -
Partie 19: Mesure des propriétés
mécaniques -
Essai de traction à température ambiante
des supraconducteurs composites de
Nb₃Sn mis en réaction
(CEI 61788-19:2013)

Supraleitfähigkeit -
Teil 19: Messung der mechanischen
Eigenschaften - Zugversuch von
reagierten Nb₃Sn-Verbundsupraleitern bei
Raumtemperatur
(IEC 61788-19:2013)

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Foreword

The text of document 90/328/FDIS, future edition 1 of IEC 61788-19, prepared by IEC/TC 90 "Superconductivity" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 61788-19:2014.

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Annex ZA (normative)

Normative references to international publications with their corresponding European publications

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN/HD</u>	<u>Year</u>
IEC 60050	series	International Electrotechnical Vocabulary	-	-
ISO 376	-	Metallic materials - Calibration of force-proving instruments used for the verification of uniaxial testing machines	EN ISO 376	-
ISO 6892-1	-	Metallic materials - Tensile testing - Part 1: Method of test at room temperature	EN ISO 6892-1	-
ISO 7500-1	-	Metallic materials - Verification of static uniaxial testing machines - Part 1: Tension/compression testing machines - Verification and calibration of the force-measuring system	EN ISO 7500-1	-
ISO 9513	-	Metallic materials - Calibration of extensometer systems used in uniaxial testing	EN ISO 9513	-

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INTRODUCTION

The Cu/Nb₃Sn superconductive composite wires are multifilamentary composite materials. They are manufactured in different ways. The first method is the bronze route, where fine Nb / Nb alloy filaments are embedded in a bronze matrix, a barrier and a copper stabilizer. The second is the internal-tin method, where fine multifilaments are composed with copper matrix including Sn reservoirs, a barrier, and a copper stabilizer. The third is the powder-in-tube method, where Nb / Nb alloy tubes are filled with Sn rich powders and are embedded in a Cu stabilizing matrix.

Common to all types of Nb₃Sn composite wires is that the superconducting A15 phase Nb₃Sn has been formed at final wire dimension by applying one or more heat treatments for several days with a temperature at the last heat treatment step of around 640 °C or above. This superconducting phase is very brittle and failure of filaments occurs – accompanied by the degradation of the superconducting properties.

Commercial composite superconductors have a high current density and a small cross-sectional area. The major application of the composite superconductors is to build superconducting magnets. This can be done either by winding the superconductor on a spool and applying the heat treatment together with the spool afterwards (wind and react) or by heat treatment of the conductor before winding the magnet (react and wind). While the magnet is being manufactured, complicated stresses are applied to its windings. Therefore the react and wind method is the minority compared to the wind and react manufacturing process.

In the case that the mechanical properties should be determined in the unreacted, non-superconducting stage of the composite, one should also apply this standard or alternatively IEC 61788-6 (*Superconductivity– Part 6: Mechanical properties measurement – Room temperature tensile test of Cu/Nb-Ti composite superconductors*).

While the magnet is being energized, a large electromagnetic force is applied to the superconducting wires because of their high current density. In the case of the react and wind manufacturing technique, the winding strain and stress levels are very restricted.

It is therefore a prerequisite to determine the mechanical properties of the superconductive reacted Nb₃Sn composite wires of which the windings are manufactured.

SUPERCONDUCTIVITY –

Part 19: Mechanical properties measurement – Room temperature tensile test of reacted Nb₃Sn composite superconductors

1 Scope

This part of IEC61788 covers a test method detailing the tensile test procedures to be carried out on reacted Cu/Nb₃Sn composite superconducting wires at room temperature.

The object of this test is to measure the modulus of elasticity and to determine the proof strength of the composite due to yielding of the copper and the copper tin components from the stress versus strain curve.

Furthermore, the elastic limit, the tensile strength, and the elongation after fracture can be determined by means of the present method, but they are treated as optional quantities because the measured quantities of the elastic limit and the elongation after fracture have been reported to be subject to significant uncertainties according to the international round robin test.

The sample covered by this test procedure should have a bare round or rectangular cross-section with an area between 0,15 mm² and 2,0 mm² and a copper to non-copper volume ratio of 0,2 to 1,5 and should have no insulation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050 (all parts), *International Electrotechnical Vocabulary* (available at <<http://www.electropedia.org>>)

ISO 376, *Metallic materials – Calibration of force-proving instruments used for the verification of uniaxial testing machines*

ISO 6892-1, *Metallic materials – Tensile testing – Part 1: Method of test at room temperature*

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

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

3 Terms and definitions

For the purposes of this document, the definitions given in IEC 60050-815 and ISO 6892-1, as well as the following, apply.

3.1
tensile stress

R
tensile force divided by the original cross-sectional area at any moment during the test

3.2
strain

A
displacement increment divided by initial gauge length of extensometers at any moment during the test

3.3
modulus of elasticity

E
gradient of the straight portion of the stress-strain curve in the elastic deformation region

3.4
extensometer gauge length

length of the parallel portion of the test piece used for the measurement of displacement by means of an extensometer

3.5
distance between grips

L_g
length between grips that hold a test specimen in position before the test is started

3.6
0,2 % proof strength

$R_{p0,2}$
stress value where the ductile components yield by 0,2 %.

Note 1 to entry: The designated proof strengths, $R_{p0,2-0}$ and $R_{p0,2-U}$ correspond to point A or point C obtained from unloading slope U between 0,3 % and 0,4 % in Figure 1(a), respectively. This strength is regarded as a representative 0,2 % proof strength of the composite.

3.7
tensile strength

R_m
tensile stress corresponding to the maximum testing force

3.8
tensile stress at elastic limit

$R_{\text{elasticmax}}$
tensile force divided by the original cross-sectional area at the transition of elastic to plastic deformation

3.9
strain at elastic limit

$A_{\text{elasticmax}}$
strain at the transition of elastic to plastic deformation

Note 1 to entry: The stress $R_{\text{elasticmax}}$ and the corresponding strain $A_{\text{elasticmax}}$ refer to point G in Figure A.6 of Annex A.5 and are regarded as the transition point of elastic to plastic deformation.

4 Principles

The test consists of straining a test piece by tensile force beyond the elastic deformation regime, in principle for the purpose of determining the modulus of elasticity (E) and the proof strengths of $R_{p0,2}$.

5 Apparatus

5.1 General

The test machine and the extensometers shall conform to ISO 7500-1 and ISO 9513, respectively. The calibration shall obey ISO 376. The special requirements of this standard are presented here.

5.2 Testing machine

A tensile machine control system that provides a constant stroke speed shall be used. Grips shall have a structure and strength appropriate for the test specimen and shall be constructed to provide a firm connection with the tensile machine. The faces of the grips shall be filed or knurled, or otherwise roughened, so that the test specimen will not slip on them during testing. Gripping may be screw type, pneumatically, or hydraulically actuated.

5.3 Extensometer

The mass of the extensometer shall be 30 g or depending on wire diameter even less, so as not to affect the mechanical properties of the brittle reacted superconductive wire. The mass of the extensometers had to be balanced symmetrically around the wire to avoid any non-alignment force (see Clause A.2). Care shall also be taken to prevent bending moments from being applied to the test specimen.

Depending on the employed strain measuring method, however, the quantities determined by the present test should be limited. When using the conventional single extensometer system, the determination of E_U and $R_{p0,2-U}$ is recommended. On the other hand, it is possible to determine all quantities described here by using an averaging double extensometer system, because of its capability to compensate the bending effects of the reacted sample and to guarantee a proper determination of the modulus of elasticity E_0 .

NOTE Further information is given in Clauses A.2 and A.3.

6 Specimen preparation

6.1 General

The wire should be straightened before heat treatment and should be inserted into a ceramic or quartz tube with slightly larger inner diameter referring to the wire size.

The constant temperature zone length of the heat treatment furnace shall be longer than the total length mentioned below in 6.2.

Care shall be taken to prevent bending or pre-loading when the reacted specimen is manually handled during removal from the ceramic or quartz tube and mounting.

6.2 Length of specimen

The total length of the test specimen shall be the sum of inward distance between grips and both grip lengths. The inward distance between grips shall be 60 mm or more, as requested for the installation of the extensometers.

6.3 Removing insulation

If the test specimen surface is coated with an insulating material, the coating shall be removed before the heat treatment. Either a chemical or mechanical method shall be used with care taken not to damage the specimen surface (see Clause A.7).

6.4 Determination of cross-sectional area (S_0)

A micrometer or other dimension-measuring apparatus shall be used to obtain the cross-sectional area of the specimen after the insulation coating has been removed. The cross-sectional area of a round wire shall be calculated using the arithmetic mean of the two orthogonal diameters. The cross-sectional area of a rectangular wire shall be obtained from the product of its thickness and width. Corrections to be made for the corners of the cross-sectional area shall be determined through consultation among the parties concerned (see Clause A.8).

7 Testing conditions

7.1 Specimen gripping

When the test specimen is mounted on the grips of the tensile machine, the test specimen and tensile loading axis shall be on a single straight line with a minimum of machine/specimen mismatch. Gripping techniques of specimen are described in Clause A.9.

7.2 Setting of extensometer

When mounting the extensometer, care shall be taken to prevent the test specimen from being deformed. The extensometer shall be mounted at the centre between the grips, aligning the measurement direction with the specimen axis direction.

During mounting care should be taken not to pre-load the specimen. After installation, loading shall be physically zeroed.

Double extensometer shall be mounted symmetrically around the cross-section to allow averaging of the strain to compensate the bending effects.

To guarantee best performance of the stress-strain curve of rectangular wires the extensometer should be mounted in such a way that strain is measured symmetrically on the small sides of the wire.

7.3 Testing speed

The tensile tests shall be performed with displacement control. The machine crosshead speed is recommended to be set between 0,1 mm/min and 0,5 mm/min.

7.4 Test

Following this procedure the tensile machine shall be started after the crosshead speed has been set to a specific level. The signals from the extensometers and the load cell shall be recorded, saved, and plotted on the abscissa and ordinate of the diagram as shown in Figures 1 (a) and 1 (b). When the total strain has reached a value between 0,3 % and 0,4 % the tensile force shall be reduced by 30 % to 40 % without changing the crosshead speed. Following this procedure the wire shall be reloaded again until final fracture.

Prior to the start of any material test program it is advisable to check the complete test equipment using similar size wires of known elastic properties (See Clause A.14).

8 Calculation of results

8.1 Modulus of elasticity (E)

Modulus of elasticity shall be calculated in general using the following formula and the straight portion of the unloading curve and of the initial loading one. Appropriate software for data evaluation should be used for post analyses of the plotted data with the possibility of enlargement of the stress versus strain graph, especially around the region where the deviation from linearity is expected.

$$E = \Delta F / (S_0 \Delta A) \quad (1)$$

where

E is the modulus of elasticity;

ΔF is the increment of the corresponding force;

ΔA is the increment of strain corresponding to ΔF ;

S_0 is the original cross-sectional area of the test specimen. Since unloading process is carried out at the strain indicated by the point A_U in Figure 1(a), the same Formula (1) is used for both the unloading modulus of elasticity (E_U) and the initial loading one (E_0). It is recommended to measure the unloading curve at the starting point A_U , where A_U is recommended to be between 0,3 % and 0,4 %.

The modulus of elasticity determined from the unloading curve is expressed as E_U which is given by the slope of the line (U between 0,3 % and 0,4 % strain) in Figure 1(a) and that from the initial loading curve is expressed as E_0 by the zero offset line.

It should be, however, noted that the straight portion of the initial stress – strain curve is very narrow as indicated in Figure A.6 of Clause A.5. To measure this quantity with a low relative standard uncertainty the only currently possible technique is the use of an averaging double extensometer system. In this sense, the quantity of E_U should be a representative data for the present text, while E_0 should be reported only when the measure is performed by means of double extensometer system.

After the test, the results shall be examined using the ratio E_0/E_U . The ratio shall satisfy the condition as given in Equation 2 in which $\Delta = 0,3$ (see Clause A.12).

$$1-\Delta < E_0/E_U < 1+\Delta \quad (2)$$

When it does not satisfy the condition, the test is judged not to be valid. Then the test shall be repeated after the experimental procedure is reexamined according to the present test method.

It is guided to achieve the unloading-reloading procedure as follows: when the loading curve arrives at the strain A_U (between 0,3 % and 0,4 %), the stress is reduced to r_{umin} of the maximum stress (stress position where the unloading started r_{umax}) and then the wire is reloaded. The slope of the unloading curves shall be obtained in the linear portion between the stress r_{umax} and r_{umin} .

NOTE 3 Typical range of r_{umax} is 99 % of the maximum stress (stress where the unloading starts). The range of r_{umin} is at 90 % referring to the onset of the unloading stress (see Figure 1 (b)).

8.2 0,2 % proof strength ($R_{p0,2-0}$ and $R_{p0,2-U}$)

The 0,2 % proof strength of the composite is determined in two ways from the unloading/reloading and initial loading part of the stress-strain curve as shown in Figures 1(a) and 1(b).

The 0,2 % proof strength of the composite under unloading $R_{p0,2-U}$ shall be determined as follows: the linear portion of the unloading slope is moved parallel to the origin of the fitted curve, which may include a negative strain value. Thereafter, a parallel line shall be shifted to 0,2 % on the abscissa from this strain point. The intersection of this line U with the stress-strain curve determines the point C that shall be defined as the 0,2 % proof strength. Depending of the unloading line (e. g. $U_{0,35}$ in Fig 1(a)), 0,2 % proof strength ($R_{p0,2-U}$) is determined.

The 0,2 % proof strength under loading $R_{p0,2-0}$ shall be determined as follows: the initial linear portion at zero offset position of the loading line of the stress-strain curve is moved 0,2 % along the strain axis and the point A at which this linear line intersects the stress-strain curve shall be defined as the 0,2 % proof strength under loading.

Each of 0,2 % proof strength value shall be calculated using the formula (3) given below:

$$R_{p0,2-i} = F_i / S_0 \quad (3)$$

where

$R_{p0,2-i}$ is the 0,2 % proof strength (MPa) at each point;

F_i is the force (N) at each point;

as $i = 0$ or U.

9 Uncertainty of measurand

Unless otherwise specified, measurements shall be carried out in a temperature that can range from 283 K to 308 K. A force measuring cell with the relative standard uncertainty less than 0,1 %, valid between zero and the maximum force capacity of load cell shall be used. The extensometers should have the relative standard uncertainty of strain less than 0,05 %. The displacement measuring transducer (e.g. LVDT [linear variable differential transformer]) used for the calibration should have the relative standard uncertainty less than 0,01 %.

The relative standard uncertainty values of measured moduli of elasticity E_0 and E_U and the proof strengths $R_{p0,2-0}$ and $R_{p0,2-U}$ currently achieved with respect to the international round robin test of eleven representative research groups are given in Table A.1 (see Clause A.12).

According to the international round robin test (see (9) of Clause A.15), the relative standard uncertainty was reported to be 1,4 % for E_0 for the test data of $N = 17$ in average after the qualification check. Similarly, 1,3 % for E_U ($N = 15$), 1,5 % for $R_{p0,2-0}$ ($N = 17$) and 2,5 % for $R_{p0,2-U}$ ($N = 13$) were reported.

10 Test report

10.1 Specimen

The following information shall be reported:

- a) Name of the manufacturer of the specimen
- b) Classification and/or symbol

c) Lot number

The following information shall be reported if possible:

- d) Raw materials and their chemical composition
- e) Cross-sectional shape and dimension of the wire
- f) Filament diameter
- g) Number of filaments
- h) Copper to non-copper ratio

10.2 Results

Results of the following mechanical properties shall be reported.

- a) Modulus of elasticity (E_0 and E_U)
- b) 0,2 % proof strengths ($R_{p0,2-0}$ and $R_{p0,2-U}$)

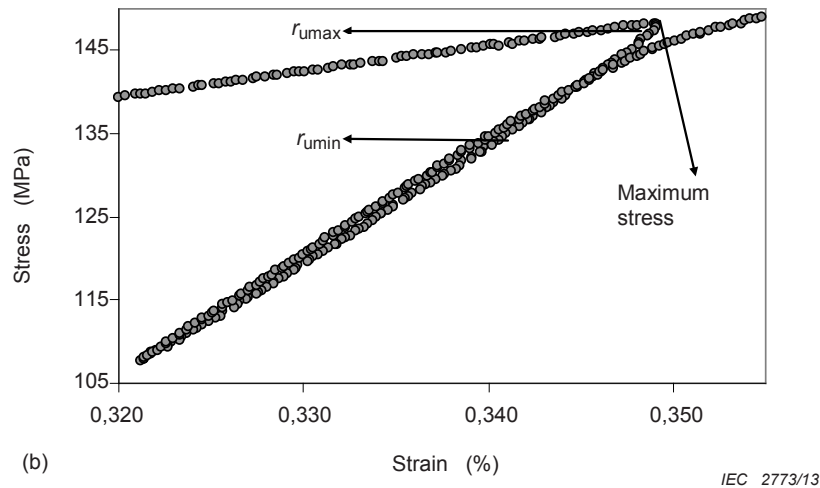
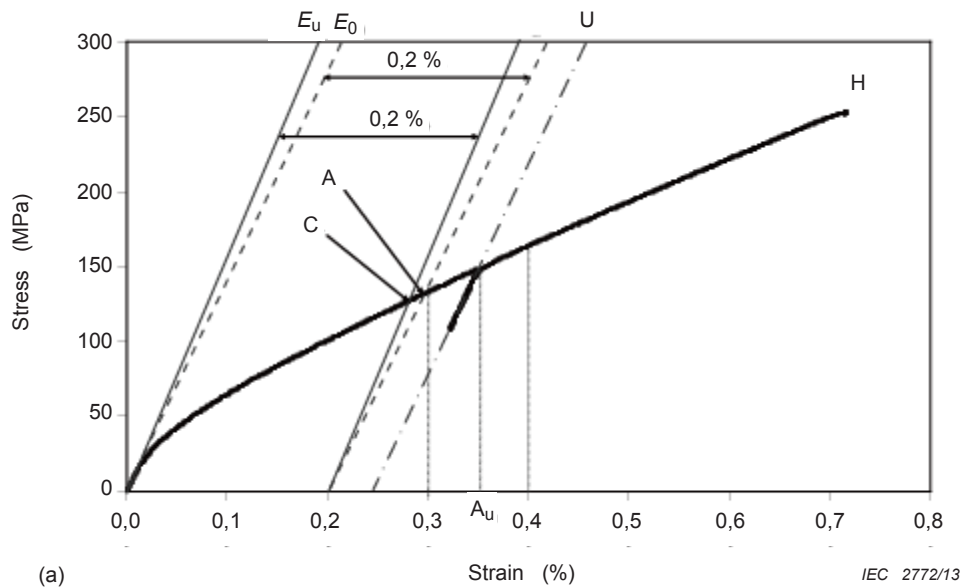
The following information shall be reported if required:

- c) Tensile stress $R_{\text{elasticmax}}$
- d) Strain $A_{\text{elasticmax}}$
- e) Tensile strength (R_m)
- f) Percentage elongation after fracture (A)
- g) 0,2 % proof strength determined by means of function fitting method ($R_{p0,2-F}$)

10.3 Test conditions

The following information shall be reported:

- a) Crosshead speed
- b) Distance between grips
- c) Temperature
- d) Manufacturer and model of testing machine
- e) Manufacturer and model of extensometers
- f) Gripping method



The Figure 1(a) shows the over-all relation between stress and strain; (b) is the enlarged view indicating the unload and reload procedure.

Key

- U: Computed unloading line of U between 0,3 % and 0,4 % strain using 1st order regression line in Figure 1(a)
- Point A: 0,2 % strain shift from initial origin of the loading line (zero offset line). $R_{p0,2-0}$ obtained experimentally.
- Point C: 0,2 % strain shift from origin of fit curve with the determined slope of unloading line U (e.g. $U_{0,35}$). $R_{p0,2-U}$ is obtained by computation.
- Point H: Final fracture point of the wire.

The slope of the initial loading line is usually smaller than that of the unloading lines. In such cases the line has to be drawn from 0,2 % offset point on the abscissa to obtain 0,2 % proof strength ($R_{p0,2-0}$) of the composite due to yielding of the ductile components such as copper and bronze (point A). Point A is obtained from the initial loading line.

Point C is obtained using the unloading line. The slope of the unloading line between 0,3 % and 0,4 % should be shifted to the origin of the fit curve, which may include a negative strain shift (see Clause A.6). The parallel 0,2 % strain shift of this slope as a line on the abscissa intersects the fitted curve at point C, which is defined as the 0,2 % proof strength of the composite ($R_{p0,2-U}$).

The graph in Figure 1(b) shows the raw data of the unloading region. The slope should be determined between 99 % of maximum stress at the onset of unloading and 90 % stress of the maximum stress as indicated (see 8.1).

Figure 1 – Stress-strain curve and definition of modulus of elasticity and 0,2 % proof strengths for Cu/Nb₃Sn wire

Annex A (informative)

Additional information relating to Clauses 1 to 10

A.1 Scope

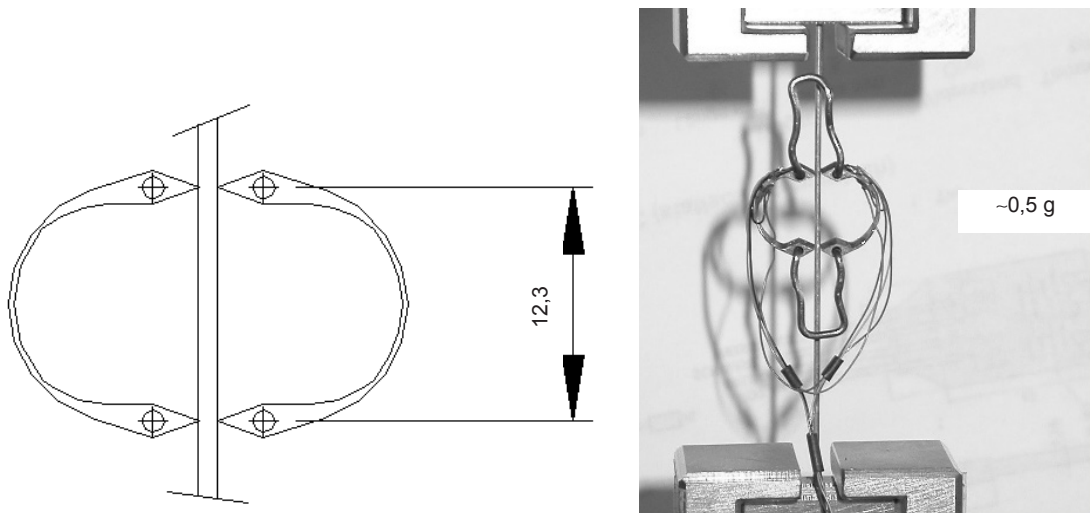
This annex gives reference information on the variable factors that may affect the tensile test methods. All items described in this annex are informative.

A.2 Extensometer

A.2.1 Double extensometer

Any type of extensometer can be used if it consists of two single extensometers capable of recording two signals to be averaged by software or one signal already averaged by the extensometer system itself.

In Figures A.1 and A.2 typical advanced light weight extensometers are shown.

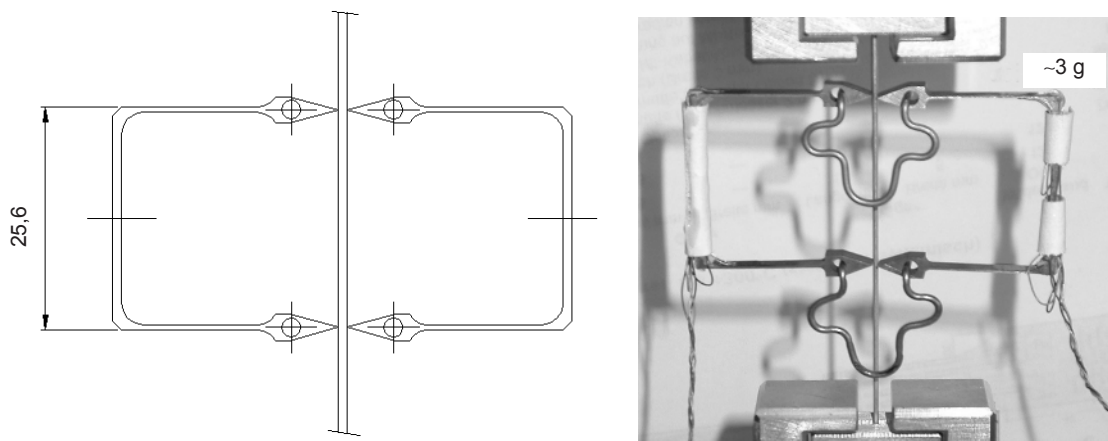


IEC 2166/13

Dimensions in millimetres

The extensometer has a gauge length of ~ 12 mm (total mass ~ 0,5 g). The two extensometers are wired together into a single type extensometer, thus averaging the two displacement records electrically.

Figure A.1 – Light weight ultra small twin type extensometer



IEC 2167/13

Dimensions in millimetres

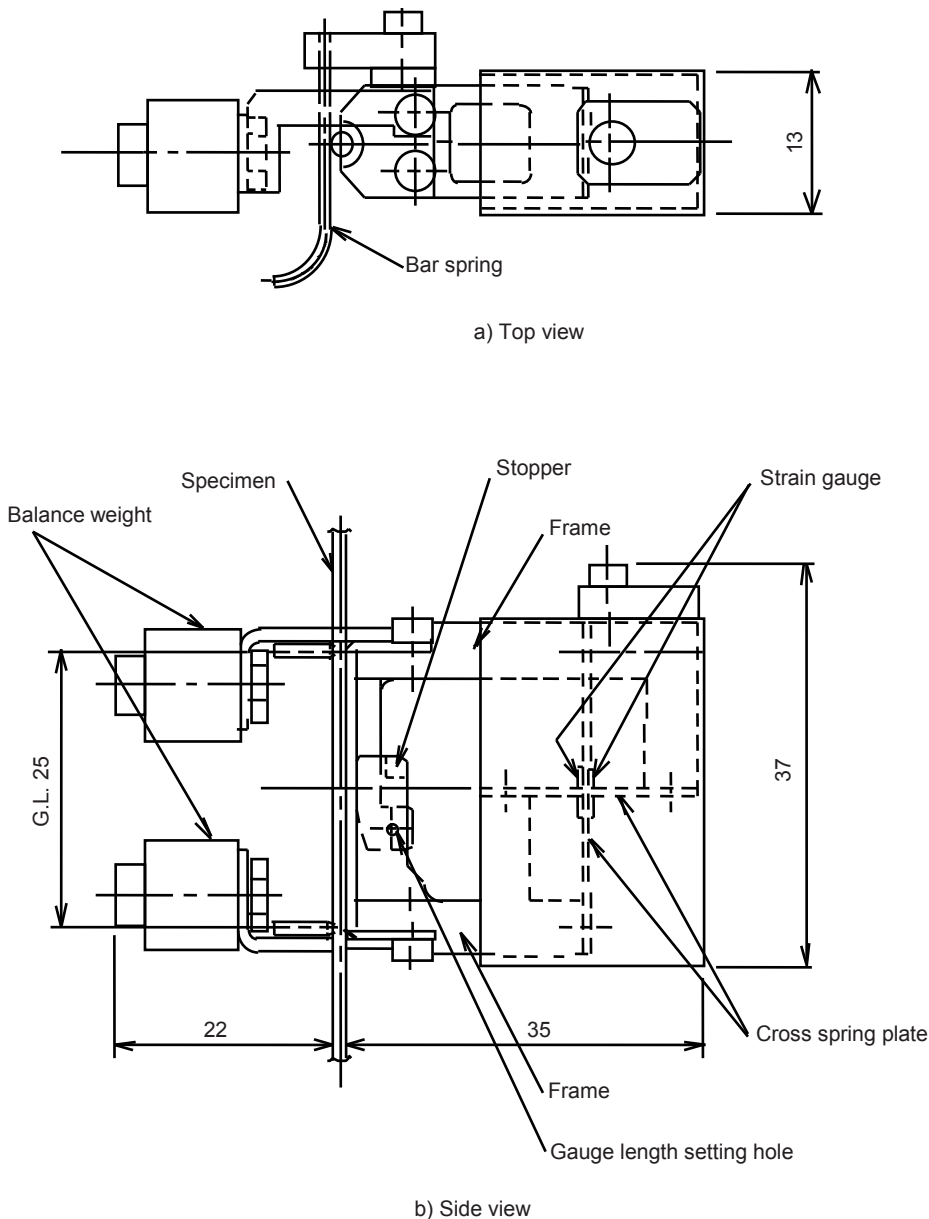
The extensometer has a gauge length of ~ 26 mm (total mass ~ 3 g). Each of the two extensometers is a single type extensometer, the averaging should be carried out by software.

Figure A.2 – Low mass averaging double extensometer

A.2.2 Single extensometer

Figure A.3 shows a single extensometer with a total weight of 31 g together with a balance weight. It was used during a RRT for Cu/Nb-Ti wires conducted in Japan and sound results were obtained. The results were used to establish the international standard (IEC 61788-6) [3, 4]¹.

¹ Figures in square brackets in this annex refer to the Reference documents listed in Clause A.15



IEC 2168/13

Dimensions in millimetres

Figure A.3 – An example of the extensometer provided with balance weight and vertical specimen axis

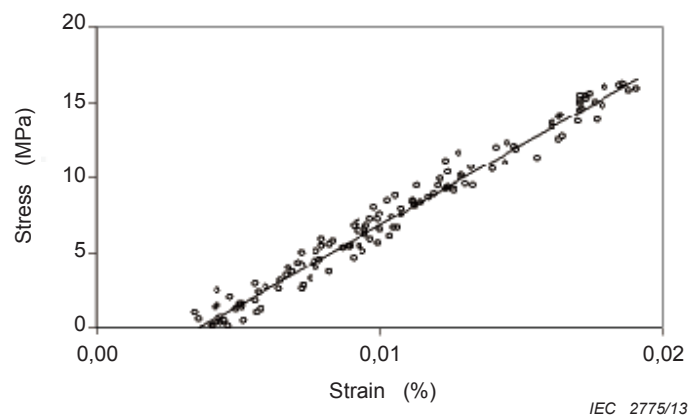
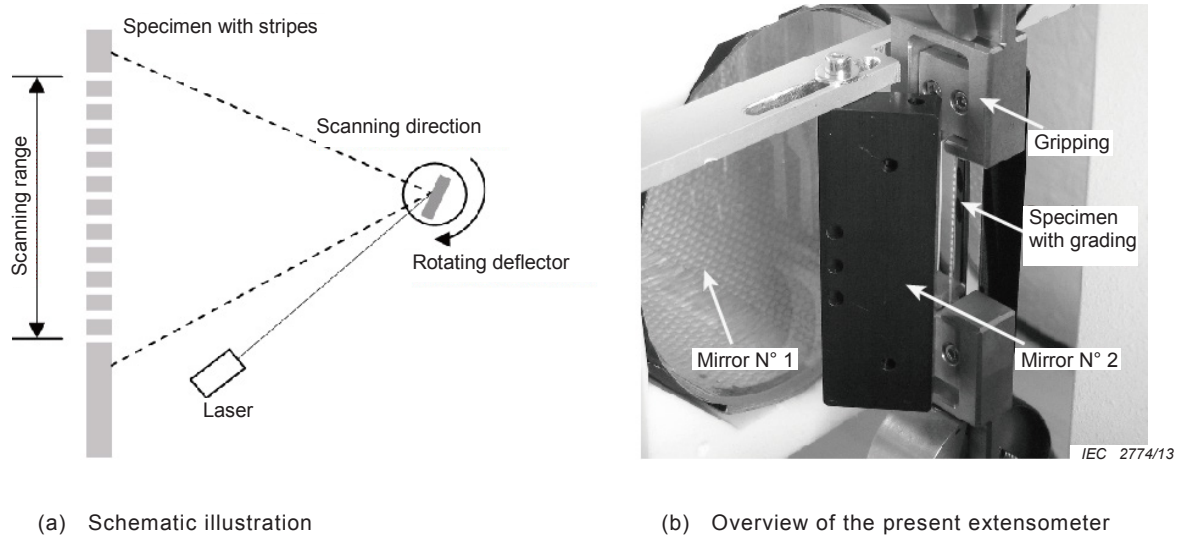
A.3 Optical extensometers

Any type of optical extensometer can be used if it is based on two single optical beams, where the signal can be recorded and averaged.

Alternatively, systems without mechanical contact to the specimen can also be used in a way similar to an averaging double extensometer system based either on two laser beams or on two other optical systems.

Figure A.4(a) shows schematically the scan of the stripes with 50 Hz of the rotating deflector. The small displacement changes during the loading of the specimen are analyzed by the

software. Figure A.5(b) shows the picture of the double mirror arrangement of a typical advanced double laser beam system.



(c) The results of a reacted Nb_3Sn wire with 0,81 mm \varnothing measured with a double beam laser extensometer.

Figure A.4 – Double beam laser extensometer

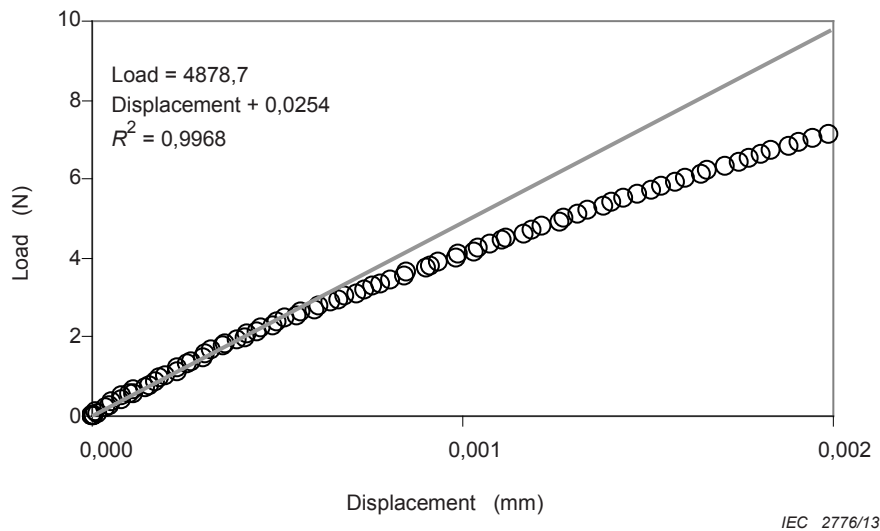
A.4 Requirements of high resolution extensometers

The requirements for such extensometers can well be derived from Figure A.5. Considering the target that the recorded values plotted from the raw data should have a low relative standard uncertainty, in particular between zero % strain and 0,01 % strain, the total displacement in this range will be 2,5 μm for the case of 25 mm gauge length or 1,2 μm for 12 mm gauge length. In fact, the signals should be acquired with a low noise around 100 times better to ensure stable records within the required strain range. The calibration factor of the used 12 mm gauge length extensometer is 10 V per 1 mm displacement. The V_{pp} of the signal should be less than 1 mV to ensure this low relative standard uncertainty. Using state-of-the-art signal conditioners, shielded and twisted cables and high resolution data acquisition systems of > 16 bit resolution, it is thus possible to ensure this demand. Figure A.5 shows the original raw data of the reacted Nb_3Sn measurement in form of load versus

displacement graph. To achieve the low scatter of the data shown it is necessary to have a high signal to noise ratio enabling to resolve the curve well below the 1 μm range [5,6,8,9]².

To obtain a zero offset gradient with a sufficient low relative standard uncertainty, which allows an assessment for the modulus of elasticity, it is prerequisite to use high resolution extensometers with extreme low noise to signal ratio.

The double extensometer system based either on two mechanical extensometers, on two laser beams, or on two other optical systems arranged symmetrically in a 180° sector to each beam may guarantee a compensation of the bending.



This figure shows the necessary low relative standard uncertainty with respect to the displacement resolution. The data are taken from the measurement of the sample as shown in Figure A.1.

Figure A.5 – Load versus displacement record of a reacted Nb₃Sn wire

A.5 Tensile stress $R_{\text{elasticmax}}$ and strain $A_{\text{elasticmax}}$

The tensile stress at which the transition of elastic to plastic deformation occurs is calculated in general using the following formula (Figure A.6).

$$R_{\text{elasticmax}} = F_{\text{elasticmax}} / S_0 \quad (\text{A.1})$$

where

$R_{\text{elasticmax}}$ is the tensile stress (MPa) at the transition of elastic to plastic deformation;

$F_{\text{elasticmax}}$ is the force (N) at the transition of elastic to plastic deformation.

The strain at which the transition of elastic to plastic deformation occurs (Figure A.6) referred to the stress $R_{\text{elasticmax}}$ is defined as follows:

$$\Delta A_{\text{total}} = A_{\text{max}} - A_0 \quad (\text{A.2})$$

² Figures in square brackets in this annex refer to the Reference documents listed in Clause A.15

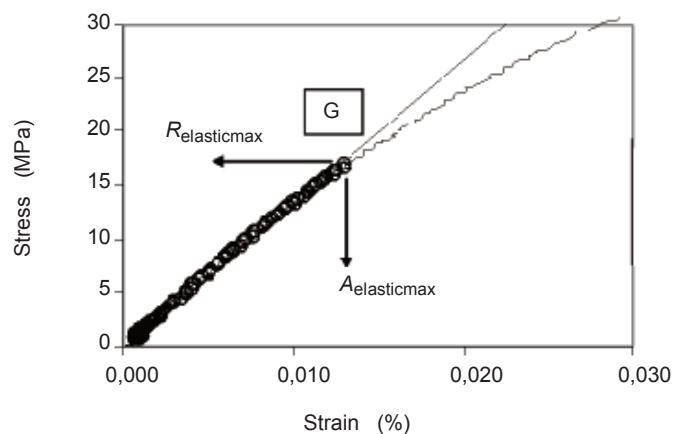
where

ΔA_{total} is the total strain increment referring to zero offset strain and to strain where the transition of elastic to plastic deformation occurs;

A_{max} is the observed value of strain referred to the stress $R_{\text{elasticmax}}$;

A_0 is the zero offset strain.

The values $R_{\text{elasticmax}}$ and $A_{\text{elasticmax}}$ are treated as being of informative character.



This is the enlarged figure up to the transition region of the elastic plastic deformation (point G). The plots (large circles) show the region of the initial straight portion of the record. The linear 1st order regression analysis gives the modulus of elasticity E ($E_0 = 134,7$ GPa) and the measure of linearity as square of regression coefficient. The value of computed 0,188 MPa is the result of the regression line analysis, where the origin has been determined to have an offset in the ordinate owing to the plot scatter. In addition, the square of regression coefficient should be greater than 0,99 to ensure the linearity. The stress $R_{\text{elasticmax}}$ and the corresponding strain $A_{\text{elasticmax}}$ correspond to the transition region of elastic plastic deformation. In particular, the value of $R_{\text{elasticmax}}$ is an important quantity for the judgment of the determined E_0 . A low value of $R_{\text{elasticmax}}$ (e.g. < 5 MPa) may indicate a higher uncertainty for the E_0 owing to the small portion of the linear region. The smaller linear range has an impact for the measurand E_0 towards high uncertainty due to the data scatter. The uncertainty may raise the question of repeating the measurement.

Figure A.6 – Stress-strain curve of a reacted Nb₃Sn wire

A.6 Functional fitting of stress-strain curve obtained by single extensometer and 0,2 % proof strength ($R_{p0,2-F}$)

The functional fitting method is applicable to determine the 0,2 % proof strength in the case of single extensometer. Usually, constitute materials of copper and bronze in the Cu/Nb₃Sn wire have been yielded during cooling from heat-treatment temperature to room temperature. The stress-strain curve, therefore, is curved from the beginning in the strict sense and the evaluation of initial modulus of elasticity becomes difficult. Furthermore, due to the non-straight form of the specimen as heat-treated condition and pre-straining on handling during setting to the tensile testing machine, the stress-strain curve is bent concave or convex, hence it is difficult to estimate the intrinsic zero strain point. The functional fitting method is effective to exclude such strain included in the experimental data. The stress-strain curves can be approximated by the following exponential function:

$$F / S_0 = a(A - b)^n \quad (\text{A.3})$$

where F , S_0 and A are load, cross section and strain obtained by the test, a , b and n are parameters determined by non-linear least-mean-square-fitting. In order to avoid losing digits

during calculation, A is expressed in %. The upper bound of fitting is 0,5 % and the lower bound of fitting is increased until the three parameters converge.

The 0,2 % proof strength $R_{p0,2-F}$ of the composite due to yielding of the copper and bronze components by function fitting method is determined as follows; the linear portion under unloading is to be moved parallel to the 0,2 % offset point with regard to the zero strain point defined by the parameter b . The intersection of this line with the fitted stress – strain curve determines the point C that is defined as the 0,2 % proof strength (Figure 1(a)). Fitting by simplified equation of (A.3) by excluding the parameter b , means neglect of pre-strain and gives larger proof stress value close to the $R_{p0,2-F}$. It is reported that commercially available non-linear least-square-fitting software can produce results almost identical to the experimental data for the same parameters, if the allowable error is selected to less than 0,1 [1,2]³. However, the confirmation of coincidence between data points and the fitting curve is made.

A.7 Removing insulation

The coating on the surface of the test specimen should be removed using an appropriate method. Normally the Nb₃Sn conductors are braided with glass or ceramic fibers which can be easily removed by stripping or peeling. In case of other type of insulation one should use either an organic solvent or a mechanical method. In both case one may do this before the heat treatment reaction and may avoid any damage of the specimen surface.

The coating is not designed as a structural component. An analysis of measurement as a multi component composite including insulation is too complicated to perform. Therefore, this test method covers only the bare reacted wire in order to maintain the mechanical behavior of the wire.

A.8 Cross-sectional area determination

In case a smaller relative standard uncertainty is required, the cross-sectional area may be obtained by correcting the radius of the corner of the rectangular wire finished by dies, using the value given on the manufacturing specifications. For rolling or Turk's-head finish, the radius of the corner is not controlled and a correction is made using a macro photograph of the cross-section.

A.9 Fixing of the reacted Nb₃Sn wire to the machine by two gripping techniques

For gripping, the specimen may be soft soldered to a metallic sleeve in the region of the grips. These sleeves should provide a firm gripping to the machine's pull rods. Alternatively a gripping can be envisaged by chucking the wire itself. In this case it should be ensured that the wire inside the chucks is not damaged mechanically. The test specimen is mounted using the grips of the tensile machine. In any case, the test specimen and tensile loading axis are aligned to disclose a mismatching. During mounting of the sample, it is necessary to prevent bending or deformation.

According to the international round robin test results, several kinds of gripping techniques were allowable to get proper test results. Therefore it is recommended that the gripping technique proposed in the present standard be treated as one of various possible techniques.

³ Figures in square brackets in this annex refer to the Reference documents listed in Clause A.15

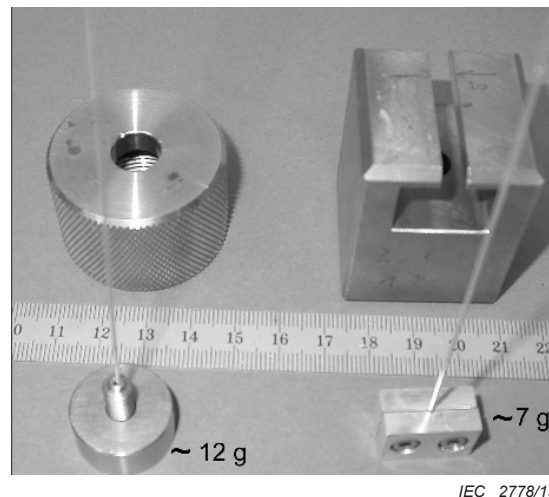
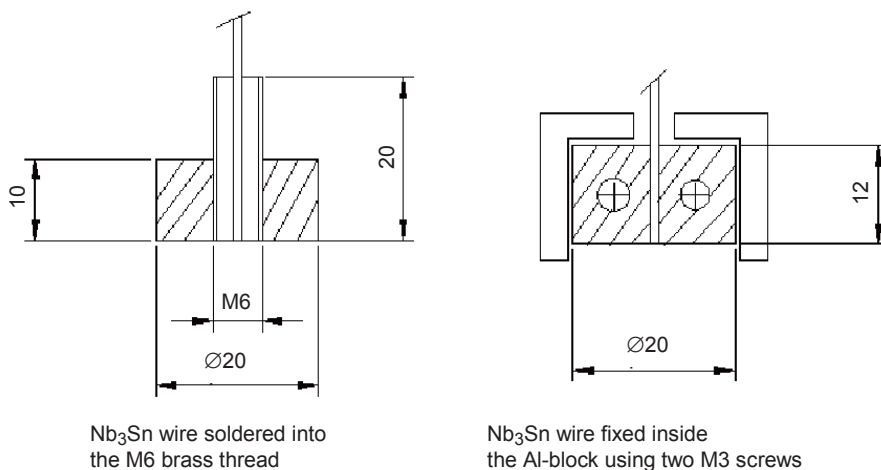


Figure A.7 – Two alternatives for the gripping technique.

The left image shows gripping of the soldered Nb₃Sn wire into a M6 brass thread, which is fixed with an aluminum sleeve serving the pulling action. The total mass of the sleeve together with the M6 thread is around 12 g.

The right portion of the image shows the clamping of the bare Nb₃Sn wire inside a V-groove of an aluminum block. The total mass of the block is around 7 g. This block inserted into a small frame acts as a defined fixture for the pulling action.



Nb₃Sn wire soldered into the M6 brass thread

Nb₃Sn wire fixed inside the Al-block using two M3 screws

IEC 2779/13

Dimensions in millimetres

The drawings show details of the two alternative possibilities of the wire fixing to the machine, by soldering and by clamping. In any case prior to the measurement start it is not allowed to load the wire with a pre-load. The bottom end of the wire with the fixing block should be free of any contact to the machine to avoid any pre-loading.

Figure A.8 – Details of the two alternatives of the wire fixing to the machine

A.10 Tensile strength (R_m)

The tensile strength at which the fracture occurs is calculated using the following formula.

$$R_m = F_{\max} / S_0 \quad (\text{A.4})$$

where

R_m is the tensile stress (MPa) at the fracture;

F_{\max} is the maximum force (N) at the fracture.

For the wire with small copper to non-copper volume ratio, premature fracture occurs at the grips giving rise to lower tensile strength and smaller percentage elongation after fracture. The tensile strength and percentage elongation after fracture are important not only from the scientific view point describing the mechanical properties of composite material, but also useful for measures of validity of the tests. However, because the variances are large and the strain region of interest for the wire is small, the values are used as references.

A.11 Percentage elongation after fracture (A_f)

The measurement of elongation after fracture may serve only as a reference. The movement of the cross-head may also be used to find the approximate value for elongation after fracture as shown below. To use this method, the cross-head position at fracture must be recorded. The following formula is used to obtain the elongation after fracture, given in percentage.

$$A_f = 100 (L_u - L_g) / L_g \quad (\text{A.5})$$

where

A_f is the percentage elongation after fracture;

L_u is the distance between grips after fracture.

L_g is the initial inward distance between grips.

A.12 Relative standard uncertainty

Owing to the nature of any measurement, the obtained test results have a scatter in all cases. To assess the quality of the measured data the concept of uncertainty serves a sound basis for an independent judgement. In Annex B and C detailed information are supplied with respect to uncertainty of a measurand.

In case of Nb₃Sn wire measurements substantial experiences were gathered during the international round robin tests carried out with 11 research groups [7,9]. In particular, the evaluation of obtained data, supplied valuable information with respect to the modulus of elasticity determined at the zero offset line and from the unloading line between 0,3 % and 0,4 % strain. In general, the obtained moduli of elasticity results from the initial loading line have a larger scatter compared to the determined values from the unloading line. A comprehensive analysis of these data is given in reference [8]⁴ and shows that the ratio of E_0 and E_U varies from unity if all results are collected together. These variation can be described by using the simple relation $1 - \Delta < E_0/E_U < 1 + \Delta$, where Δ defines the quantity of the deviation from unity. To limit the scatter at the initial loading line and to delete disqualified data $\Delta = 0,3$ has been proven to be an adequate value.

In Table A.1 the important quantities such as the modulus of elasticity obtained from initial loading line and also the proof strengths are summarized. The standard uncertainties of these results show that the disqualification of the data beyond $\Delta = 0,3$ reduces the standard uncertainties at least by a factor of 2.

⁴ Figures in square brackets in this annex refer to the Reference documents listed in Clause A.15

The scatter of experimental data obtained from RRT relates to two contributions of the intra- and inter- laboratory. In order to make clear whether the data submitted from their respective laboratories belongs into the same population of all experimental data, the analysis of variance (F-test) was performed under the guidance of the text (GUM H.5.2.1). As a typical example, the experimental data on E_0 were analysed and the results are listed in Table A.1. In order to make exact comparison among laboratories, it is necessary to fix the same number of data from each laboratory, three for example. So three data were chosen randomly from each laboratory when the number of data was equal to or larger than 3, while the data set from laboratory was abandoned in case of a data number less than 3.

According to GUM guidance, the ratio of the inter-laboratory variance (s_a^2) and the intra-laboratory one (s_b^2) was calculated as $F_{\text{exp}} = s_a^2 / s_b^2$ and compared with the theoretical function, $F_{N-J}^{J-1}(\alpha)$. When the hypothesis of $F_{\text{exp}} < F_{N-J}^{J-1}(\alpha)$ holds, the data belong to the same population with the significance level α %. In the case, where all data were employed for F-test, the hypothesis did not hold for some samples, E3, E4, H and M. On the other hand, in another case, where the data qualified by applying Δ of 0,3, were used, the hypothesis held for all the samples, when α was settled on 1 %. Therefore it was concluded that the present F-test guarantees the validity of qualification check with respect to the ratio of moduli of elasticity mentioned in 8.1 of the main text. Further it is possible to judge the qualification condition more rigidly by using the higher significance level.

Table A.1 – Standard uncertainty value results achieved on different Nb₃Sn wires during the international round robin tests

Sample	Property(X)	All Data				Qualified Data			
		N	<x>	SU	RSU	N'	<x'>	SU	RSU
E2	E_0 , [GPa]	35	113,5 [GPa]	2,8[GPa]	2,5[%]	22	114 [GPa]	1,4 [GPa]	0,3[%]
	$R_{p0,2-0}$, [MPa]	35	187,1 [MPa]	3,0[MPa]	1,6[%]	22	181,7 [MPa]	1,3 [MPa]	0,7[%]
E3	E_0 , [GPa]	33	119,2[GPa]	3,2 [GPa]	2,7[%]	21	121[GPa]	1,5[GPa]	1,3[%]
	$R_{p0,2-0}$, [MPa]	34	192,1[MPa]	2,3 [MPa]	1,2[%]	21	191,8 [MPa]	1,6 [MPa]	0,8[%]
E4	E_0 , [GPa]	36	90,3[GPa]	3,9[GPa]	4,3[%]	14	109,2[GPa]	2,0[GPa]	1,9[%]
	$R_{p0,2-0}$, [MPa]	37	113,6[MPa]	2,2 [MPa]	1,9[%]	14	111,9 [MPa]	3,7 [MPa]	3,3[%]
H	E_0 , [GPa]	33	88,5[GPa]	4,2 [GPa]	4,8[%]	9	109,8 [GPa]	2,0 [GPa]	1,8[%]
	$R_{p0,2-0}$, [MPa]	33	118,8[MPa]	2,3[MPa]	1,9[%]	9	110,3[MPa]	2,9[MPa]	2,6[%]
K	E_0 , [GPa]	34	116,2[GPa]	2,8 [GPa]	2,4[%]	25	115,6 [GPa]	1,1 [GPa]	1,0[%]
	$R_{p0,2-0}$, [MPa]	33	182[MPa]	2,6 [MPa]	1,4[%]	25	179,7 [MPa]	2,6 [MPa]	1,4[%]
M	E_0 , [GPa]	29	88,6[GPa]	5,8 [GPa]	6,6[%]	9	120,4[GPa]	3,0[GPa]	2,5[%]
	$R_{p0,2-0}$, [MPa]	28	118 [MPa]	4,0 [MPa]	3,4[%]	9	109,2[MPa]	1,7[MPa]	1,5[%]

N: number of total tested wires,
N': number of qualified wires,
X: modulus of elasticity or proof strength,
<x>: average for total wires,
<x'>: average for qualified wires,
SU: standard uncertainty for total data, and
RSU: relative standard uncertainty.

This table presents results of standard uncertainty values achieved on different Nb₃Sn wires during the international round robin tests carried out by 11 different research laboratories.

Consequently, the average value of relative standard uncertainty (U_{RSU}) for all samples is given by the equation

$$\overline{U_{\text{RSU}}} = \frac{\sum_{m=1}^M N'_m U_{\text{RSU}m}}{\sum_{m=1}^M N'_m} \quad (\text{A6})$$

where M indicates the number of different wires and the average number of samples is given,

$$\overline{N'} = \frac{\sum_{m=1}^M N'_m}{M} \quad (\text{A7})$$

Then $\overline{U_{\text{RSU}}}$ was calculated from Table A.1 as 1,5 % for E_0 for $\overline{N'} = 17$ as the average value for all samples after the qualification check. By using the same procedure, the $\overline{U_{\text{RSU}}}$ was evaluated for E_u , $R_{p0.20}$ and $R_{p0.2U}$ and their result is presented in Clause 9 of the main text.

Table A.2 – Results of ANOVA (F-test) for the variations of E_0

Sample	All data					Qualified data				
	J	N	s_a^2/s_b^2	F_{exp}	$F_{N-1}^{J-1}(\alpha)$	J'	N'	s_a^2/s_b^2	F_{exp}	$F_{N-1}^{J-1}(\alpha)$
E2	8	24	318 / 195	1,6	4,05	8	24	318 / 195	1,6	4,05
E3	9	27	803 / 163	4,9	3,73	8	24	500 / 182	2,7	4,05
E4	9	27	1189 / 165	7,2	3,73	7	21	613 / 205	2,9	4,46
H	9	27	1388 / 180	7,6	3,73	6	18	841 / 238	3,5	5,06
K	9	27	205 / 96	2,1	3,73	9	27	205 / 96	2,1	3,73
M	8	24	3180 / 191	16,5	4,05	3	9	912 / 158	5,7	10,9

The significance level of 1% was used for the verification of the hypothesis. Every data set from a laboratory includes three values ($n = 3$) and therefore the following relation holds as $N = nJ$. Here, J : number of laboratories, N : number of wires in total, J' : number of qualified laboratories, N' : number of qualified wires.

A.13 Determination of modulus of elasticity E_0

The determination of modulus of elasticity E_0 requires a data acquisition system allowing a low relative standard uncertainty at zero-offset regime of the stress-strain record. To ensure unbiased data the recorded data of stress and strain should be evaluated according to the following recommended procedure.

Using the stress versus strain original record one may determine the first order linear regression line and the square of the regression coefficient between zero MPa and 50 MPa stresses. Within this context the control parameter is the square of the regression coefficient which should be greater than 0,99. By stepwise reducing the stress starting from the upper value 50 MPa, try to determine the linear slope fulfilling this condition. This linear slope is defined to be the modulus of elasticity E_0 . The intersection of the linear slope with the abscissa is the new origin of the stress versus strain graph, which is considered during the 0,2 % parallel shift for the estimation of the $R_{p0.2-0}$ value (see Figure 1).

A.14 Assessment on the reliability of the test equipment

The reliability of the test equipment, which comprises the tensile testing unit, load cell and the used extensometer system, can be best analyzed with wires of similar sizes and known elastic properties. Around one mm diameter welding wires of the materials aluminium and pure commercial titanium have been approved to be the most suitable ones, which cover the modulus of elasticity range between 70 GPa and 100 GPa. It is strongly recommended that the test laboratory confirm the reliability of its tensile setup from time to time by measuring the elastic properties of these wires prior to any measurement task. These wires can be easily purchased from vendors. For these tests the wires should be handled in the same manner as described for the case with superconducting heat treated wires. The wire can be loaded and unloaded in elastic regime up to 100 MPa without affecting its elastic properties.

A.15 Reference documents

- 1) *Research report on the standardization of superconductive materials for new power generation system*. NMC, Osaka Science and Technology Center, 2001, 23
- 2) M. SHIMADA, M.HOJO, H.MORIAI and K.OSAMURA. *Jpn. Cryogenic Eng.*, 33, 1998, 665
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- 4) *Research report on the standardization of superconductive materials for new power generation system*. NMC, Osaka Science and Technology Center, 2002, 25
- 5) K.OSAMURA, A.NYILAS, M.SHIMADA, H.MORIAI, M.HOJO, T.FUSE and M.SUGANO. *Adv. Superconductivity XI*, 1999, 1515
- 6) A. NYILAS. Strain sensing systems tailored for tensile measurement of fragile wires. *Supercond. Sci. Technol.*, 18, 2005, 409 – 415
- 7) A. NYILAS, K. WEISS, and M. THOENER, M. HOJO, K. OSAMURA, and K. KATAGIRI. On the measurement of tensile properties of superconducting Nb₃Sn wires at ambient temperature and at cryogenic environment. *Advances in Cryogenic Engineering (Materials) 52*, edited by U. B.Balachandran et al., Plenum Press, New York, 2006, 582 – 589
- 8) A. NYILAS, Transducers for sub-micron displacement measurements at cryogenic temperatures. in *Advances in Cryogenic Engineering (Materials) 52*, edited by U. B.Balachandran et al., Plenum Press, New York, 2006, 27 – 34
- 9) K. OSAMURA et al. International Round Robin Test for Mechanical Properties of Nb₃Sn Superconducting Wires. *Supercond. Sci. Technol.*, 21, 2008, 045006

Annex B (informative)

Uncertainty considerations

B.1 Overview

In 1995, a number of international standards organizations, including IEC, decided to unify the use of statistical terms in their standards. It was decided to use the word “uncertainty” for all quantitative (associated with a number) statistical expressions and eliminate the quantitative use of “precision” and “accuracy.” The words “accuracy” and “precision” could still be used qualitatively. The terminology and methods of uncertainty evaluation are standardized in the Guide to the Expression of Uncertainty in Measurement (GUM) [1]⁵.

It was left to each TC to decide if they were going to change existing and future standards to be consistent with the new unified approach. Such change is not easy and creates additional confusion, especially for those who are not familiar with statistics and the term uncertainty. At the June 2006 TC 90 meeting in Kyoto, it was decided to implement these changes in future standards.

Converting “accuracy” and “precision” numbers to the equivalent “uncertainty” numbers requires knowledge about the origins of the numbers. The coverage factor of the original number may have been 1, 2, 3, or some other number. A manufacturer’s specification that can sometimes be described by a rectangular distribution will lead to a conversion number of $1/\sqrt{3}$. The appropriate coverage factor was used when converting the original number to the equivalent standard uncertainty. The conversion process is not something that the user of the standard needs to address for compliance to TC 90 standards, it is only explained here to inform the user about how the numbers were changed in this process. The process of converting to uncertainty terminology does not alter the user’s need to evaluate their measurement uncertainty to determine if the criteria of the standard are met.

The procedures outlined in TC 90 measurement standards were designed to limit the uncertainty of any quantity that could influence the measurement, based on the Convener’s engineering judgment and propagation of error analysis. Where possible, the standards have simple limits for the influence of some quantities so that the user is not required to evaluate the uncertainty of such quantities. The overall uncertainty of a standard was then confirmed by an interlaboratory comparison.

B.2 Definitions

Statistical definitions can be found in three sources: the GUM, the International Vocabulary of Basic and General Terms in Metrology (VIM)[2], and the NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (NIST)[3]. Not all statistical terms used in this standard are explicitly defined in the GUM. For example, the terms “relative standard uncertainty” and “relative combined standard uncertainty” are used in the GUM (5.1.6, Annex J), but they are not formally defined in the GUM (see [3]).

B.3 Consideration of the uncertainty concept

Statistical evaluations in the past frequently used the coefficient of variation (COV) which is the ratio of the standard deviation and the mean (N.B. the COV is often called the relative

⁵ Figures in square brackets refer to the reference documents in Clause B.5 of this annex.

standard deviation). Such evaluations have been used to assess the precision of the measurements and give the closeness of repeated tests. The standard uncertainty (SU) depends more on the number of repeated tests and less on the mean than the COV and therefore in some cases gives a more realistic picture of the data scatter and test judgment. The example below shows a set of electronic drift and creep voltage measurements from two nominally identical extensometers using same signal conditioner and data acquisition system. The $n = 10$ data pairs are taken randomly from the spreadsheet of 32 000 cells. Here, extensometer number one (E_1) is at zero offset position whilst extensometer number two (E_2) is deflected to 1 mm. The output signals are in volts.

Table B.1 – Output signals from two nominally identical extensometers

Output signal [V]	
E_1	E_2
0,001 220 70	2,334 594 73
0,000 610 35	2,334 289 55
0,001 525 88	2,334 289 55
0,001 220 70	2,334 594 73
0,001 525 88	2,334 594 73
0,001 220 70	2,333 984 38
0,001 525 88	2,334 289 55
0,000 915 53	2,334 289 55
0,000 915 53	2,334 594 73
0,001 220 70	2,334 594 73

Table B.2 – Mean values of two output signals

Mean (\bar{X}) [V]	
E_1	E_2
0,001 190 19	2,334 411 62

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n} \quad [\text{V}] \quad (\text{B.1})$$

Table B.3 – Experimental standard deviations of two output signals

Experimental standard deviation (s) [V]	
E_1	E_2
0,000 303 48	0,000 213 381

$$s = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^n (X_i - \bar{X})^2} \quad [\text{V}] \quad (\text{B.2})$$

Table B.4 – Standard uncertainties of two output signals

Standard uncertainty (<i>u</i>) [V]	
<i>E</i> ₁	<i>E</i> ₂
0,000 095 97	0,000 067 48

$$u = \frac{s}{\sqrt{n}} \quad [V] \quad (B.3)$$

Table B.5 – Coefficient of Variations of two output signals

Coefficient of variation (COV) [%]	
<i>E</i> ₁	<i>E</i> ₂
25,498 2	0,009 1

$$COV = \frac{s}{X} \quad (B.4)$$

The standard uncertainty is very similar for the two extensometer deflections. In contrast the coefficient of variation *COV* is nearly a factor of 2 800 different between the two data sets. This shows the advantage of using the standard uncertainty which is independent of the mean value.

B.4 Uncertainty evaluation example for TC 90 standards

The observed value of a measurement does not usually coincide with the true value of the measurand. The observed value may be considered as an estimate of the true value. The uncertainty is part of the "measurement error" which is an intrinsic part of any measurement. The magnitude of the uncertainty is both a measure of the metrological quality of the measurements and improves the knowledge about the measurement procedure. The result of any physical measurement consists of two parts: an estimate of the true value of the measurand and the uncertainty of this "best" estimate. The GUM, within this context, is a guide for a transparent, standardized documentation of the measurement procedure. One can attempt to measure the true value by measuring "the best estimate" and using uncertainty evaluations which can be considered as two types: Type A uncertainties (repeated measurements in the laboratory in general expressed in the form of Gaussian distributions) and Type B uncertainties (previous experiments, literature data, manufacturer's information, etc. often provided in the form of rectangular distributions).

The calculation of uncertainty using the GUM procedure is illustrated in the following example:

- a) The user must derive in the first step a mathematical measurement model in the form of identified measurand as a function of all input quantities. A simple example of such model is given for the uncertainty of a force, *F*_{LC} measurement using a load cell:

$$F_{LC} = W + d_W + d_R + d_{Re}$$

where *W*, *d*_W, *d*_R, and *d*_{Re} represent the weight of standard as expected, the manufacturer's data, repeated checks of standard weight/day and the reproducibility of checks at different days, respectively.

Here the input quantities are: the measured weight of standard weights using different balances (Type A), manufacturer's data (Type B), repeated test results using the digital electronic system (Type B), and reproducibility of the final values measured on different days (Type B).

b) The user should identify the type of distribution for each input quantity (e.g. Gaussian distributions for Type A measurements and rectangular distributions for Type B measurements).

c) Evaluate the standard uncertainty of the Type A measurements,

$u_A = \frac{s}{\sqrt{n}}$ where, s is the experimental standard deviation and n is the total number of measured data points.

d) Evaluate the standard uncertainties of the Type B measurements:

$u_B = \sqrt{\frac{1}{3} \cdot d_W^2 + \dots}$ where, d_W is the range of rectangular distributed values

e) Calculate the combined standard uncertainty for the measurand by combining all the standard uncertainties using the expression:

$$u_c = \sqrt{u_A^2 + u_B^2}$$

In this case, it has been assumed that there is no correlation between input quantities. If the model equation has terms with products or quotients, the combined standard uncertainty is evaluated using partial derivatives and the relationship becomes more complex due to the sensitivity coefficients [4, 5].

f) Optional – the combined standard uncertainty of the estimate of the referred measurand can be multiplied by a coverage factor (e. g. 1 for 68 % or 2 for 95 % or 3 for 99 %) to increase the probability that the measurand can be expected to lie within the interval.

g) Report the result as the estimate of the measurand \pm the expanded uncertainty, together with the unit of measurement, and, at a minimum, state the coverage factor used to compute the expanded uncertainty and the estimated coverage probability.

To facilitate the computation and standardize the procedure, use of appropriate certified commercial software is a straightforward method that reduces the amount of routine work [6, 7]. In particular, the indicated partial derivatives can be easily obtained when such a software tool is used. Further references for the guidelines of measurement uncertainties are given in [3, 8, and 9].

B.5 Reference documents of Annex B

- [1] ISO/IEC Guide 98-3:2008, *Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement* (GUM 1995)
- [2] ISO/IEC Guide 99:2007, *International vocabulary of metrology – Basic and general concepts and associated terms* (VIM)
- [3] TAYLOR, B.N. and KUYATT, C.E. *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*. NIST Technical Note 1297, 1994 (Available at <<http://physics.nist.gov/Pubs/pdf.html>>)
- [4] KRAGTEN, J. Calculating standard deviations and confidence intervals with a universally applicable spreadsheet technique. *Analyst*, 1994, 119, 2161-2166
- [5] EURACHEM / CITAC Guide CG 4 Second edition:2000, *Quantifying Uncertainty in Analytical Measurement*
- [6] Available at <http://www.gum.dk/e-wb-home/gw_home.html>
- [7] Available at <<http://www.isgmax.com/>>

- [8] CHURCHILL, E., HARRY, H.K., and COLLE,R., *Expression of the Uncertainties of Final Measurement Results*. NBS Special Publication 644 (1983)
- [9] JAB NOTE Edition 1:2003, *Estimation of Measurement Uncertainty (Electrical Testing / High Power Testing)*. (Available at:
<<http://www.iaac.org.mx/Documents/Uncontrolled/Library/JapanAccredBoard/nws-lab-topix.pdf>>).

Annex C (informative)

Specific examples related to mechanical tests

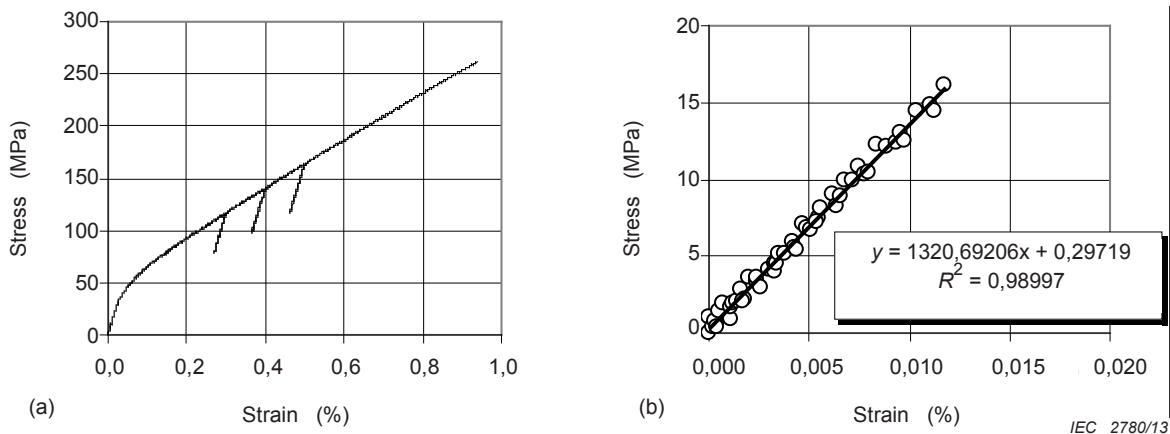
C.1 Overview

These are specific examples to illustrate techniques of uncertainty estimation. The inclusion of these examples does not imply that users must complete a similar analysis to comply with the standard. However, the portions that estimate the uncertainty of each individual influence quantity (load, displacement, wire diameter, and gauge length) need to be evaluated by the user to determine if they meet the specified uncertainty limits in the standard.

These two examples are not meant to be exhaustive. They do not include all possible sources of error, such as friction, bent/straightened wire, removal of insulation, misaligned grips, and strain rate. These additional sources may or may not be negligible.

C.2 Uncertainty of the modulus of elasticity

In Figure C.1, the original stress versus strain raw data of a Nb₃Sn wire (diameter 0,768 mm) is given. These measurements were carried out during the course of an international round robin test in 2006. Figure C.1 (a) shows the loading of the wire up to fracture, while Figure C.1 (b) displays points taken during the initial loading up to 16 MPa and the line fit to these data. The computed slope of the trend line is 132069 MPa (the slope is expand with a factor of 100 due to unit percentage of abscissa) as given in Figure 1 (b) with a squared correlation coefficient of 0,9899.



Graph (a) shows the measured stress versus strain curve of the 0,783 mm diameter superconducting wire. Graph (b) shows the initial part of the curve and the regression analysis to determine modulus of elasticity. The slope of the line should be multiplied by 100 to convert the percentage strain to strain, so that the units of modulus of elasticity will be MPa.

Figure C.1 – Measured stress-strain curve

The standard uncertainty estimation of modulus of elasticity for this wire can be processed in following way. The modulus of elasticity determined during mechanical loading is a function of five variables each having its own specific uncertainty contribution.

$$E = f(P, \Delta L, D, L_G, b), \tag{C.1}$$

The model equation is

$$E = \frac{4 \cdot P \cdot L_G}{\pi \cdot D^2 \cdot \Delta L} + b \quad (\text{C.2})$$

where

E = modulus of elasticity, MPa

P = load, N

ΔL = deflected length of extensometer in zero offset region for the selected load portion, mm

D = diameter of wire, mm

L_G = length of extensometer at start of the loading, mm

b = an estimate of deviation from the experimentally obtained modulus of elasticity, MPa.

The actual experimental values are necessary for the standard uncertainty calculation. Using the data of Figure C.1 (b) the value of deflected extensometer length can be estimated. Here, a stress of 15 MPa is selected and by using the calculated modulus of elasticity given in Figure C.1 (b) the value of ΔL can be established using the equations,

$$A = \frac{R}{E} \quad \text{and} \quad \Delta L = A \cdot L_G \quad (\text{C.3})$$

where

$$A = 1,136 \times 10^{-4}$$

$$\Delta L = 1,363 \times 10^{-3} \text{ mm}$$

$$R = 15 \text{ MPa}$$

$$L_G = 12 \text{ mm}$$

$$D = 0,783 \text{ mm}$$

Furthermore, with

$$P = \frac{\pi \cdot D^2 \cdot R}{4} \quad (\text{C.4})$$

the force P can be calculated as $P = 7,223 \text{ N}$.

C.3 Evaluation of sensitivity coefficients

The combined standard uncertainty associated with model Equation (2) is:

$$u_c = \sqrt{\left(\frac{\partial E}{\partial P}\right)^2 u_1^2 + \left(\frac{\partial E}{\partial \Delta L}\right)^2 u_2^2 + \left(\frac{\partial E}{\partial D}\right)^2 u_3^2 + \left(\frac{\partial E}{\partial L_G}\right)^2 u_4^2 + \left(\frac{\partial E}{\partial b}\right)^2 u_5^2} \quad (\text{C.5})$$

The partial differential terms are the so-called sensitivity coefficients. By substituting the experimental values in each derivative, the sensitivity coefficients c_i can be calculated as follows:

$$\text{For } c_1: \quad c_1 = \frac{\partial}{\partial P} \left(\frac{4 \cdot L_G \cdot P}{\pi \cdot D^2 \cdot \Delta L} \right) = \frac{4 \cdot L_G}{\pi \cdot D^2 \cdot \Delta L} = 1,829 \times 10^4 \text{ mm}^{-2} \quad (\text{C.6})$$

$$\text{For } c_2: \quad c_2 = \frac{\partial}{\partial \Delta L} \left(\frac{4 \cdot L_G \cdot P}{\pi \cdot D^2 \cdot \Delta L} \right) = \frac{-4 \cdot L_G \cdot P}{\pi \cdot D^2 \cdot \Delta L^2} = -9,69 \times 10^7 \text{ N} \cdot \text{mm}^{-3} \quad (\text{C.7})$$

$$\text{For } c_3: \quad c_3 = \frac{\partial}{\partial D} \left(\frac{4 \cdot L_G \cdot P}{\pi \cdot D^2 \cdot \Delta L} \right) = \frac{-8 \cdot L_G \cdot P}{\pi \cdot D^3 \cdot \Delta L} = -3,373 \times 10^5 \text{ N} \cdot \text{mm}^{-3} \quad (\text{C.8})$$

$$\text{For } c_4: \quad c_4 = \frac{\partial}{\partial L_G} \left(\frac{4 \cdot L_G \cdot P}{\pi \cdot D^2 \cdot \Delta L} \right) = \frac{4 \cdot P}{\pi \cdot D^2 \cdot \Delta L} = 1,101 \times 10^4 \text{ N} \cdot \text{mm}^{-3} \quad (\text{C.9})$$

Sensitivity coefficient c_5 is unity (1) owing to the differentiation of Equation 2 with respect to quantity b .

Using the above sensitivity coefficients, the combined standard uncertainty u_c is finally given by:

$$u_c = \sqrt{(c_1)^2 \cdot (u_1)^2 + (c_2)^2 \cdot (u_2)^2 + (c_3)^2 \cdot (u_3)^2 + (c_4)^2 \cdot (u_4)^2 + (c_5)^2 \cdot (u_5)^2} \quad (\text{C.10})$$

where the square of each sensitivity coefficient is multiplied by the square of the standard uncertainty of individual variables as given in the model Equation (C.2).

C.4 Combined standard uncertainties of each variable

The standard uncertainties u_i in Equation (C.10) are the combined standard uncertainties of force (P), deflected length (ΔL), wire diameter (D), and gauge length (L_G). In this section, each combined standard uncertainty will be estimated according to the available data.

The combined standard uncertainty u_1 for force P is composed of statistical distributions of Type A and Type B. In general, the force is measured with commercially available load cells. The bulk of load cell manufacturers, however, do not give information about uncertainties in their specifications. The given accuracies, along with other information obtained from the data sheets, must be first converted into standard uncertainties prior to the determination of combined standard uncertainty u_1 . Typically these manufacturer's specifications are viewed as limits to a rectangular distribution of errors. The standard uncertainty associated with the rectangular distribution is the limit divided by $\sqrt{3}$.

For the measurements given in Figure C.1, the following information for the load cell was available.

Table C.1 – Load cell specifications according to manufacturer's data sheet

Load cell capacity	Accuracy class tension / compression	Temperature coefficient of zero	Temperature coefficient of sensitivity	Creep for 30 minutes
N	%	S %/K	S %/K	S%
5 000	0,25	0,25	0,07	0,07

According to this specification, the data should be converted to standard uncertainty values before combining them. These data are treated as Type B uncertainties. The temperature range between 303 K and 283 K ($\Delta T = 20$ K) has been selected to reflect allowable laboratory conditions.

The variables are as follows:

Accuracy class:	$T_{\text{class}} = 0,25 \%$
Temperature coefficient of zero balance:	$T_{\text{coefzero}} = (0,25 \times 20) \%$
Temperature coefficient of sensitivity:	$T_{\text{coefsens}} = (0,07 \times 20) \%$
Creep for 30 min:	$T_{\text{creep}} = 0,07 \%$

The following equation describes the measurement of load and includes the four sources of error from Table C.1:

$$P = u_P + T_{\text{class}} + T_{\text{coefzero}} + T_{\text{coefsens}} + T_{\text{creep}} \quad (\text{C.11})$$

where u_P is the true value of load.

The percentage specifications are converted to load units based on the measured value of $P = 7,223 \text{ N}$ obtained from the stress versus strain curve. The resulting values are converted to standard uncertainties assuming a rectangular distribution so that the combined standard uncertainty for the load cell is:

$$u_1 = \sqrt{\left(\frac{T_{\text{class}} \cdot 7,223}{100 \cdot \sqrt{3}}\right)^2 + \left(\frac{T_{\text{coefzero}} \cdot 7,223}{100 \cdot \sqrt{3}}\right)^2 + \left(\frac{T_{\text{coefsens}} \cdot 7,223}{100 \cdot \sqrt{3}}\right)^2 + \left(\frac{T_{\text{creep}} \cdot 7,223}{100 \cdot \sqrt{3}}\right)^2} \quad (\text{C.12})$$

$$u_1 = 0,21 \text{ N} \quad (\text{C.13})$$

Tables C.2-C.4 summarize uncertainty calculations for displacement, wire diameter, and gauge length. These calculations are similar to those previously demonstrated for force.

Table C.2 – Uncertainties of displacement measurement

Extensometer displacement, mm	Type A Gaussian distribution. Creep and noise contribution $u_A = s / \sqrt{n}$ according Clause B.3 $2 V = 1 \text{ mm}$ ($0,0003 \sqrt{1/2} / \sqrt{10}$) mm	Type B distribution obtained from data scatter of Figure 1(b) $u_B = d_w / \sqrt{3}$ according Clause B.4 with d_w of $0,00003$ mm
$1,363 \times 10^{-4}$	0,00005	0,000017
$u_2 = \sqrt{0,00005^2 + 0,00017^2} = 0,000052 \text{ mm}$		

Table C.3 – Uncertainties of wire diameter measurement

Wire diameter, mm	Type A Gaussian distribution. Five repeated measurements with micrometer device $u_A = s / \sqrt{n}$ $(0,0013) / \sqrt{5}$ mm	Half width of rectangular distribution according manufacture data sheet accuracy of $\pm 4 \mu\text{m}$ $u_B = d_W / \sqrt{3}$ mm
0,783	0,00058	0,0023
$u_3 = \sqrt{0,00058^2 + 0,0023^2} = 0,0023 \text{ mm}$		

To measure the gauge length of the extensometer, a stereo microscope was used with a resolution of $20 \mu\text{m}$.

Table C.4 – Uncertainties of gauge length measurement

Gauge length, mm	Type A Gaussian distribution. Five repeated measurements with micrometer device $u_A = s / \sqrt{n}$ $(0,002) / \sqrt{5}$ mm	Half width of rectangular distribution according manufacture data sheet accuracy of $\pm 20 \mu\text{m}$ $u_B = d_W / \sqrt{3}$ mm
12	9×10^{-4}	0,011
$u_4 = \sqrt{0,0009^2 + 0,011^2} = 0,011 \text{ mm}$		

Finally, the uncertainty in the slope of the fitted stress versus strain curve given in Figure C.1 (b) is estimated. The maximum half width difference between the measured stress values and the calculated stress values using the trend line equation from Figure C.1 (b) result in $\pm 0,822 \text{ MPa}$. Using this value with gauge length ($L_G = 12 \text{ mm}$) and extensometer deflection value ($\Delta L = 0,001363 \text{ mm}$), a Type B uncertainty for the modulus of elasticity can be estimated. Rearranging Equation (C.3) results in the simple equation:

$$R = E \cdot A; \quad E = R \cdot \frac{L_G}{\Delta L} \quad (\text{C.14})$$

The Type B uncertainty of the measured modulus of elasticity of the Figure C.1 (b) is

$$u_b = \frac{0,822 \text{ MPa} \cdot 12 \text{ mm}}{0,00136 \text{ mm} \cdot \sqrt{3}} = 4180 \text{ MPa} \quad (\text{C.15})$$

The final combined standard uncertainty, taking into account the result of Equation (C.12) and using the sensitivity coefficients for the four variables in Equation (C.10), results in:

$$u_c = \sqrt{(1,829 \cdot 10^4)^2 \cdot (0,21)^2 + (-9,69 \cdot 10^7)^2 \cdot (0,0000521)^2 + (-3,373 \cdot 10^5)^2 \cdot (0,0023)^2 + (1,101 \cdot 10^4)^2 \cdot (0,011)^2 + (1)^2 \cdot (4180)^2} \quad (\text{C.16})$$

$$u_c = 7\,630 \text{ MPa} \quad (\text{C.17})$$

or

$$E = 132 \text{ GPa} \pm 7,6 \text{ GPa} \quad (\text{C.18})$$

C.5 Uncertainty of 0,2 % proof strength $R_{p0,2}$

The 0,2 % proof strength $R_{p0,2}$ should be determined by the parallel shifting of the modulus of elasticity zero offset line to the 0,2 % strain position along the abscissa and computing the intersection of this line with the original stress versus strain curve. If the fitted modulus of elasticity line has a different origin than zero, the offset from zero should be also considered. The regression equation in Figure C.1 (b) has an x-axis offset of:

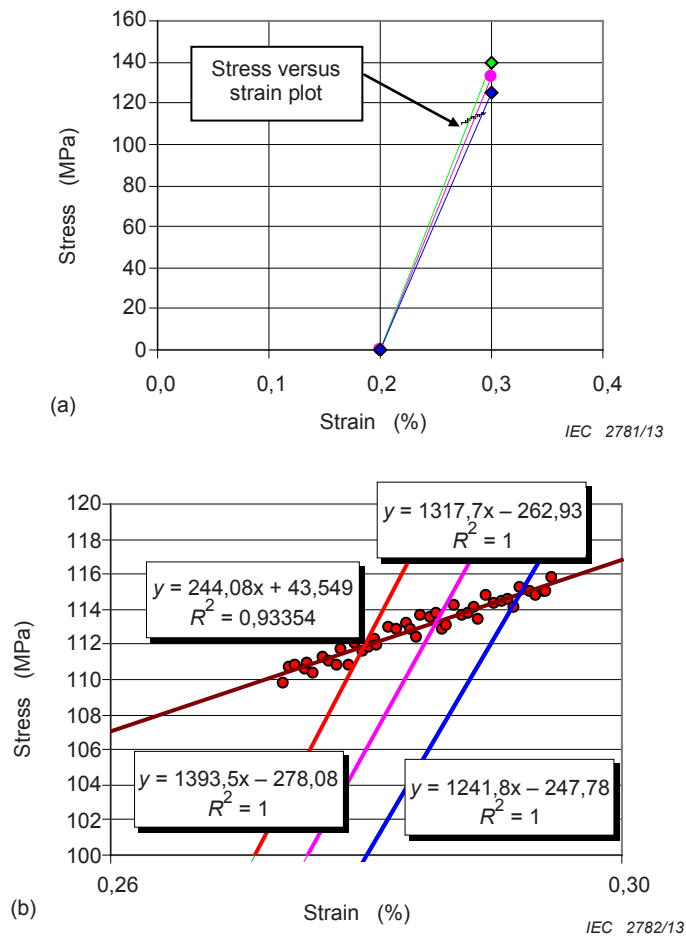
$$A_{\text{offset}} = -\frac{0,29719}{1320,692} = -0,00023 \text{ \%} \quad (\text{C.19})$$

where A_{offset} indicates offset strain at zero stress.

Thus, the shifted position of the line along the abscissa is not exactly 0,20000 % but 0,19977 %. Table C.5 shows the computation of stress using the regression line with and without the uncertainty contribution from Equation (C.18).

Table C.5 – Calculation of stress at 0 % and at 0,1 % strain using the zero offset regression line as determined in Figure C.1 (b)

Description	Regression line equation with uncertainty contribution at ε % strain	Stress at $A = 0$ % strain, MPa	Stress at $A = 0,1$ % strain, MPa
Baseline modulus of elasticity	$1320,692 \cdot A + 0,29719$	0,297	132,37
Modulus of elasticity with + 7,6 GPa uncertainty contribution (upper line)	$1396,692 \cdot A + 0,29719$	0,297	139,97
Modulus of elasticity with – 7,6 GPa uncertainty contribution (lower line)	$1244,692 \cdot A + 0,29719$	0,297	124,77



Graph (a) shows the 0,2 % offset shifted regression line and the two lines using plus and minus uncertainty contributions relative to the base line. Four points are necessary to construct the three lines; one common point at zero stress and three calculated stress values at 0,1 % strain as shown in Table C.5, however, the corresponding strain values need to be shifted by 0,2 %. In graph (a) the raw stress versus strain curve is also plotted around the region where the three lines intersect the raw data. Graph (b) shows the original raw data of stress versus strain in an enlarged view and the shifted lines according to the computations of Table C.5. The linear regression equations of all four functions are also given in this graph (b).

Figure C.2 – Stress-strain curve

In Table C.5 the selected stresses at 0 % strain and at 0,1 % strain are arbitrarily chosen for the purpose of obtaining two distinct points to determine the shifted lines in Figure C.2. The offset shift value obtained from Equation (C.19) is added to the values of 0 % strain and 0,1 % strain.

Table C.6 lists the linear regression equations after shifting the lines as determined in Figure C.2 (b).

Table C.6 – Linear regression equations computed for the three shifted lines and for the stress–strain curve in the region where the lines intersect

Description of equations	Linear regression equation. x is here the strain in % and y the stress in MPa
Linear part of stress versus strain curve (see Figure C.2 a)	$y = 244,08 \cdot x + 43,546$
Shifted modulus of elasticity baseline	$y = 1317,7 \cdot x - 262,93$
Modulus of elasticity with + 7,6 GPa uncertainty contribution (shifted upper line)	$y = 1393,5 \cdot x - 278,08$
Modulus of elasticity with – 7,6 GPa uncertainty contribution (shifted lower line)	$y = 1241,8 \cdot x - 247,78$

Finally, using the equations of Table C.6, the three intersection points are computed and the stresses at these points are determined. Table C.7 shows the computation and resulting intersection values. The reported value of proof strength is the stress of the intersection of the first line (shifted zero offset) with the stress versus strain curve. The remaining two values of stress at the intersection represent estimated error bounds for the proof strength. The error bounds are based on the uncertainty of the modulus of elasticity slope (Equation (C.18)).

Table C.7 – Calculation of strain and stress at the intersections of the three shifted lines with the stress–strain curve

Description	Equation set for strain and stress calculation at intersections	Strain at intersection, %	Stress at intersection, MPa
Shifted baseline (mean)	$(43,546 + 262,93) / (1317,7 - 244,08)$	0,285463	
	$244,08 \cdot 0,285463 + 43,546$		113,2
Shifted upper line	$(43,546 + 247,78) / (1241,8 - 244,08)$	0,291995	
	$244,08 \cdot 0,291995 + 43,546$		114,8
Shifted lower line	$(43,546 + 278,08) / (1393,5 - 244,08)$	0,279819	
	$244,08 \cdot 0,279819 + 43,546$		111,8

The standard uncertainty of the proof strength is a Type B determination, and can be estimated using:

$$\text{Uncertainty Type B: } u_b = \frac{114,82 - 111,84}{\sqrt{3}} = 0,858 \text{ MPa} \quad (\text{C.20})$$

The scatter of the raw data shown in Figure C.2 (b) should also be considered in the final uncertainty estimate. Table C.8 shows the measured stress versus strain data of Figure C.2 (b). In addition, columns 3 and 7 of Table C.8 give the computed stress using the linear fit to the data in the region of interest. Finally, columns 4 and 8 show the differences between measured and computed data.

Table C.8 – Measured stress versus strain data and the computed stress based on a linear fit to the data in the region of interest

Strain %	Stress MPa	Calculated according regression equation MPa	Difference calculated observed MPa	Strain %	Stress MPa	Calculated according regression equation MPa	Difference calculated observed MPa
0,2736	109,74	110,3219	0,5819	0,2844	113,63	112,9699	-0,6601
0,2740	110,73	110,4081	-0,3219	0,2850	113,51	113,1374	-0,3726
0,2744	110,81	110,5288	-0,2812	0,2856	113,71	113,2778	-0,4322
0,2752	110,56	110,7209	0,1609	0,2860	112,89	113,3838	0,4938
0,2755	110,90	110,7801	-0,1199	0,2863	113,08	113,4577	0,3777
0,2759	110,36	110,8958	0,5358	0,2869	114,23	113,5858	-0,6442
0,2766	111,29	111,0560	-0,2340	0,2875	113,69	113,7483	0,0583
0,2772	111,02	111,2062	0,1862	0,2881	113,77	113,8887	0,1187
0,2778	110,78	111,3466	0,5666	0,2885	114,08	113,9799	-0,1001
0,2781	111,75	111,4353	-0,3147	0,2888	113,38	114,0636	0,6836
0,2786	110,75	111,5634	0,8134	0,2894	114,79	114,2213	-0,5687
0,2791	112,08	111,6718	-0,4082	0,2900	114,29	114,3666	0,0766
0,2797	111,62	111,8294	0,2094	0,2907	114,47	114,5341	0,0641
0,2803	111,83	111,9600	0,1300	0,2912	114,59	114,6499	0,0599
0,2807	112,27	112,0782	-0,1918	0,2917	114,07	114,7731	0,7031
0,2809	111,94	112,1250	0,1850	0,2921	115,23	114,8888	-0,3412
0,2817	113,00	112,3221	-0,6779	0,2928	115,01	115,0564	0,0464
0,2824	112,86	112,4970	-0,3630	0,2933	114,81	115,1795	0,3695
0,2832	113,14	112,6743	-0,4657	0,2939	115,03	115,3273	0,2973
0,2835	112,86	112,7606	-0,0994	0,2941	114,97	115,3790	0,4090

The extreme differences between the computed and measured stress from the 4th and 8th columns of Table C.8 are:

$$-0,6779 \text{ MPa and } + 0,8134 \text{ MPa} \quad (\text{C.21})$$

The extreme differences represent observed limits to random error which can be converted to a standard uncertainty using:

$$\text{Uncertainty Type B: } u_b = \frac{0,8134 - (-0,6779)}{2\sqrt{3}} = 0,4305 \quad (\text{C.22})$$

Combined standard uncertainty for 0, 2 % proof strength is given:

$$\text{Combined uncertainty: } u_c = \sqrt{0,858^2 + 0,4305^2} = 0,96 \text{ MPa} \quad (\text{C.23})$$

Thereafter, the 0,2 % proof strength result is given as:

0,2 offset proof strength: $R_{p0,2} = 113,2\text{MPa} \quad +/\text{-} \quad 0,96\text{MPa}$ (C.24)

Bibliography

ASTM E 83-85, *Standard Practice for Verification and Classification of Extensometers*

ASTM E 111-82, *Standard Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus*

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