BS ISO 6892-4:2015

BSI Standards Publication

Metallic materials — Tensile testing

Part 4: Method of test in liquid helium

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National foreword

This British Standard is the UK implementation of ISO 6892-4:2015. It supersedes BS ISO 19819:2004 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/101/1, Uniaxial testing.

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

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Metallic materials — Tensile testing —

Part 4:
Method of test in liquid helium

Matériaux métalliques — Essai de traction —

Partie 4: Méthode d'essai dans l'hélium liquide

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Contents

Page

Foreword

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The committee responsible for this document is ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 1, *Uniaxial testing*.

This first edition of ISO 6892-4 cancels and replaces ISO 19819:2004, which has been technical revised.

ISO 6892 consists of the following parts, under the general title *Metallic materials — Tensile testing*:

— *Part 1: Method of test at room temperature*

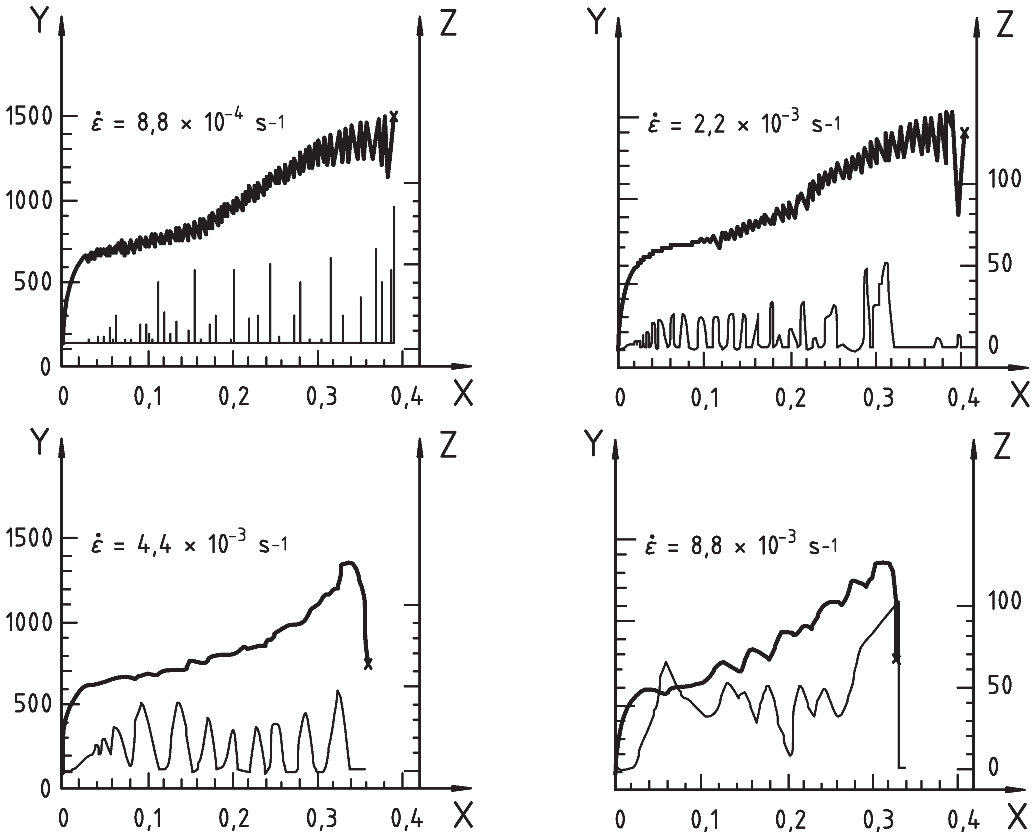
— *Part 2: Method of test at elevated temperature*

— *Part 3: Method of test at low temperature*

— *Part 4: Method of test in liquid helium*

Introduction

The force-time and force-extension records for alloys tested in liquid helium using displacement control are serrated. Serrations are formed by repeated bursts of unstable plastic flow and arrests. The unstable plastic flow (discontinuous yielding) is a free-running process occurring in localized regions of the parallel length at higher rates than nominal strain rates with internal test piece heating. Examples of serrated stress-strain curves for a typical austenitic stainless steel with discontinuous yielding are shown in Figure 1.



Key

1 stress, N/mm2

2 strain

3 temperature, K

Figure 1 — Example of typical stress-strain curves and test piece temperature histories at four different nominal strain rates, for AISI 304L stainless steel tested in liquid helium

A constant test piece temperature cannot be maintained at all times during testing in liquid helium. Due to adiabatic heating, the test piece temperature at local regions in the parallel length rises temporarily above 4 K during each discontinuous yielding event (see Figure 1). The number of events and the magnitude of the associated force drops are a function of the material composition and other factors such as test piece size and test speed. Typically, altering the mechanical test variables can change the type of serration but not eliminate the discontinuous yielding. Therefore, tensile property measurements of alloys in liquid helium (especially tensile strength, elongation, and reduction of area) may lack the usual significance of property measurements at room temperature where deformation is more nearly isothermal and discontinuous yielding typically does not occur.

Strain control is the preferred control mode (Method A, 6892-1) and displacement control is the secondary method, according to Method B 6892-1.

Metallic materials — Tensile testing —

Part 4:
Method of test in liquid helium

1 Scope

This part of ISO 6892 specifies the method of tensile testing of metallic materials in liquid helium (the boiling point is –269 °C or 4,2 K, designated as 4 K) and defines the mechanical properties that can be determined.

This part of ISO 6892 may apply also to tensile testing at cryogenic temperatures (less than –196 °C or 77 K), which requires special apparatus, smaller test pieces, and concern for serrated yielding, adiabatic heating, and strain-rate effects.

To conduct a tensile test according to this part of ISO 6892 at 4 K, the test piece installed in a cryostat is fully submerged in liquid helium (He) and tested using displacement control at a nominal strain rate of 10^{-3} s^{-1} or less.

NOTE The boiling point of the rare ^{3}He isotope is 3,2 K. Usually, the tests are performed in ^{4}He or a mixture of ^{3}He and ^{4}He with a high concentration of ^{4}He. Therefore, the temperature is, as designated before, 4 K.

2 Normative references

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

ISO 6892-1:—[1)], *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

ISO 6892-3, *Metallic materials — Tensile testing — Part 3: Method of test at low temperature*

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

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

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 6892-1 and in ISO 6892-3 apply.

3.1
adiabatic heating
internal heating of a test piece resulting from deformation under conditions such that the heat generated by plastic work cannot be quickly dissipated to the surrounding cryogen

1) To be published.

3.2
axial strain
longitudinal strains measured at opposite or equally spaced surface locations on the sides of the longitudinal axis of symmetry of the test piece

Note 1 to entry: The longitudinal strains are measured using two or more strain-sensing transducers located at the mid-length of the parallel length.

3.3
bending strain
difference between the strain at the surface of the test piece and the axial strain

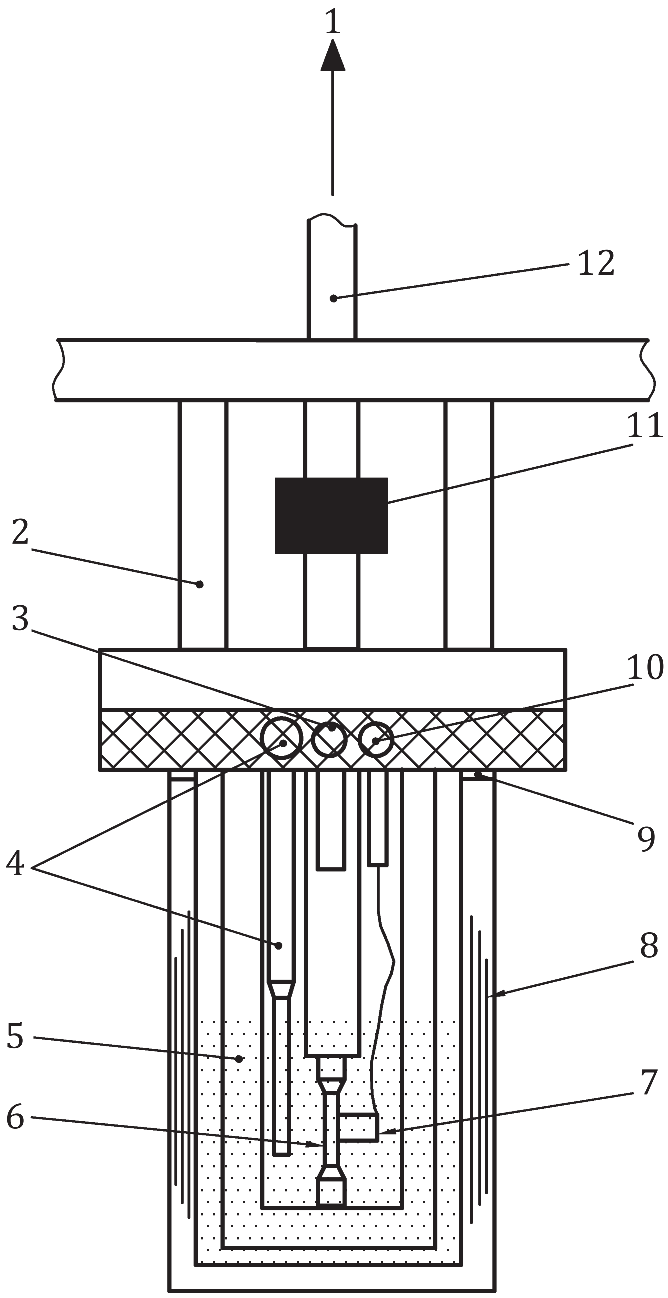
Note 1 to entry: The bending strain varies around the circumference and along the parallel length of the test piece.

3.4
dewar
vacuum-insulated container for cryogenic fluids

3.5
discontinuous yielding strength
R_i
peak stress at the initiation of the first measurable serration on the stress-strain curves

3.6
tensile cryostat
test apparatus for applying tensile forces to test pieces in cryogenic environments

Note 1 to entry: See Figure 2.



Key

1	force	7	extensometer
2	room temperature load frame	8	vacuum-insulated dewar
3	vent	9	dewar seal
4	vacuum-insulated transfer tube	10	electrical feed-through
5	cryogenic load frame	11	load cell
6	test piece	12	pull rod

Figure 2 — Schematic illustration of typical cryostat for tensile testing at 4 K

4 Symbols and designations

Symbols and corresponding designations are given in Table 1.

Table 1 — Symbols and designations

Symbol	Unit	Designation
d_o	mm	diameter of the parallel length of a cylindrical test piece or diameter of a circular wire
L_o	mm	original gauge length
L_u	mm	final gauge length after fracture
L_c	mm	parallel length
L_e	mm	extensometer gauge length
S_o	mm^2	original cross-sectional area of the parallel length
S_u	mm^2	minimum cross-sectional area after fracture (final cross-sectional area)
Z	%	percentage reduction of area: $Z = \frac{S_o - S_u}{S_o} \times 100$
A	%	percentage elongation after fracture: $A = \frac{L_u - L_o}{L_o} \times 100$
F_m	N	maximum force
R_m	N/mm^2	tensile strength
$R_{p0,2}$	N/mm^2	0,2 % proof strength, plastic extension
R_i	N/mm^2	discontinuous yielding strength

5 Principle

The test consists of straining a test piece in liquid helium by a tensile force, generally to fracture, for the purpose of determining one or more of the mechanical properties defined in Clause 4.

6 Apparatus

6.1 Testing machine

The testing machine shall be verified and calibrated in accordance with ISO 7500-1 and shall be of at least class 1, unless otherwise specified in the product standard.

6.1.1 Testing machine compliance

Compliance (displacement per unit of applied force of the apparatus itself) of the test facility (tensile machine and the cryogenic load frame) should be known. Measure the compliance by coupling the load train with a rigid test piece or by using a special calibration test piece. Then, measure the compliance at a low force and at the highest force used to qualify the machine, as indicated in 6.1.4. A practical procedure for the determination of the compliance respective of the stiffness is described in ISO 6892-1:—, Annex F.

NOTE Different system compliances may result in different stress-extension curves and material properties (e.g. elongation after fracture, tensile strength) of the material because a larger discontinuous deformation occurs in a lower compliance test facility.

6.1.2 System design

Typically, alloys in liquid helium exhibit double or triple their ambient strengths at ambient temperature. For the same test piece geometry, higher forces shall be applied to the cryostat, test piece, load train members, and grips at cryogenic temperatures. Since many conventional test machines have a maximum force of 100 kN or less, it is recommended that the apparatus be designed to accommodate one of the small test pieces cited in 7.2.

6.1.3 Construction materials

Many construction materials, including the vast majority of ferritic steels, are brittle at 4 K. To prevent service failures, fabricate the grips and other load train members using strong, tough, cryogenic alloys. Materials that have low thermal conductivity are desirable to reduce heat flow. Austenitic stainless steels (AISI 304LN), maraging steels (200, 250, or 300 grades, with nickel plating to prevent rust), wrought nickel-base superalloys, and titanium alloys (Ti-6Al-4V and Ti-5Al-2,5Sn) have been used with proper design, for grips, pull rods, and cryostat frames. Non-metallic materials (for example, glass-epoxy composites) are excellent insulators and are sometimes used for compression members.

6.1.4 Alignment

Proper system alignment is essential to minimize bending strains in the tensile tests. The machine and grips should be capable of applying force to a precisely machined calibration test piece so that the maximum bending strain should be according to ISO 23788 class 10. Reduce bending strain to an acceptable level by making proportional adjustments to a cryostat with alignment capability, or by using spacing shims to compensate an unadjustable fixture. Calculate the strain based on readings taken while the calibration test piece is subjected to a low force, as well as at the highest force for which the machine and load train are being qualified.

Qualify the apparatus by making axiality measurements at room temperature and at 4 K. To perform axiality tests of the apparatus, the test piece form and cryostat should be the same as that used during cryogenic tests, and the test piece concentricity should be as nearly perfect as possible. No plastic strain should occur in the parallel length of the alignment test piece during loading. In some cases this may necessitate the use of a relatively stiff, high-strength calibration test piece.

For cylindrical test pieces, calculate the maximum bending strain defined in 4.3 from the strains measured with three electrical-resistance strain gauges, extensometers, or clip gauges at circumferential positions, equally spaced around and at the centre of the parallel length of the test piece.

For test pieces of square or rectangular cross-section, measure the strain at the centre of two parallel (opposite) faces, or in the case of thin cross-sections, at the centre of the two broad faces.

For conventional threaded or pinned grips, evaluate the effect of test piece bias as follows. Repeat the axiality measurements with the test piece rotated 180°, but with the grips and pull rods retained in their original positions. Then calculate the maximum bending strain and the strain at the test piece axis. If other grips or methods are used to evaluate the effect of test piece bias it should be described in the report.

Strain-Averaging Technique - Nonaxiality of loading (which may be introduced due to the machining of the test pieces) is usually sufficient to introduce errors in tensile tests at small strains when strain is measured at only one position on the test piece. Therefore, measure strains at three equally spaced (or, if good alignment has been achieved, at least two opposing) positions within the parallel section. Report the average of the strains from the two or three positions centred on the parallel length.

6.1.5 Gripping mechanisms

Choose the gripping mechanism according to test piece type. Cryogenic materials shall be used in the construction of components to avoid failure in service.

6.2 Cryostats and support apparatus

6.2.1 Cryostats

In general, cryostat load frames for existing test machines are custom-built and designed to accommodate commercially available dewars. The cryostat may employ adjustable load columns to facilitate alignment.

6.2.2 Dewars

A dewar capable of retaining liquid helium is required. Stainless steel dewars are safer (that is, more fracture resistant) than glass. Generally, a single helium dewar (see Figure 2) is sufficient for short-term tensile tests. Also possible is a double-dewar arrangement in which an outer-jacketed dewar of liquid nitrogen surrounds the inner dewar of liquid helium.

6.2.3 Ancillary Equipment

Dewars and transfer lines for liquid helium shall be vacuum insulated. Vacuum pumps, pressurized gas, and liquid nitrogen facilities are therefore required.

6.3 Liquid-level indicators

Maintaining a liquid helium environment ensures the intended test condition. With the test piece completely immersed, a thermocouple to measure its temperature is not required for routine tests. Instead, a simple indicator or meter is required to ensure that the test piece remains fully submerged throughout the test. An on-off indicator of the carbon-resistor type located at some reference point in the cryostat may be used to verify that the liquid level always remains above the test piece. Alternatively, the liquid level may be continuously monitored using a superconducting wire sensor of appropriate length positioned vertically inside the cryostat.

6.4 Extensometer

6.4.1 Types

Reliable clip-gauge extensometers for use at 4 K may be purchased or built. When using extensometers to measure the extension, the extensometers shall be of class 1 (see ISO 9513) in the relevant range for the determination of the 0,2 % proof strength; for the determination of other properties (corresponding to higher elongations) the extensometers of class 2 (see ISO 9513) in the relevant range may be used.

To determine the proof strength, two or more extensometers shall be used. Whenever possible, mount the extensometer(s) knife edges directly to the test piece parallel length.

High excitation voltage on the extensometers can create a noisy strain signal due to the creation localized boiling on the grid of the strain gauges due to heating of the strain gauge grid. In order to prevent influence of heating, the bridge voltage should be lowered to approximately 1 V.

NOTE As long as the temperature of the gauge remains constant during the test and the voltage is not high enough to cause boiling on the grid, gauge heating should not be an issue.

Extensometers that use capacitance measurement to monitor strain may be used. The type with overlapping concentric cylinders has an extended strain range. Use the linear portion of the displacement with adjustable sensitivity.

Strain gauges bonded directly to the test piece surface may be used to measure strain at 4 K. When using the strain gauge at cryogenic temperature, care shall be taken for the selection and combination of gauge materials, base materials, and adhesives. However, it should be considered that strain gauges might de-bond before reaching the 0,2 % proof strength .

6.4.2 Calibration

Calibrate extensometers at room temperature and at 4 K. For calibrations at 4 K, a device such as a micrometer with vertical extension tubes can be used with the extensometer(s) mounted at the lower end and immersed in liquid helium. If the calibration is known and proved to be accurate, linear, and reproducible, then room-temperature checks may be performed before each series of tests to indirectly verify the 4 K calibration. However, direct calibration at 4 K shall be performed periodically, especially if damage is suspected or repairs have been made.

7 Test piece

7.1 General

The shape and dimensions of the test pieces depend on the shape and dimensions of the metallic products of which the mechanical properties are to be determined.

7.2 Standard round bar test piece

To meet the force limitations of conventional test machines, the round bar test piece with a 7 mm diameter and a gauge length (L_o)/diameter (d) ratio of 5 is defined as standard for 4 K tests. Threaded or shouldered ends are common for gripping these test pieces, and the requirement of 6.1.4 can be met by precise machining.

7.3 Alternatives

If the standard test piece described above is inappropriate for some reason, other sizes and other shapes of the cross-section may be selected. Wire is a special case of a round bar test piece with a small diameter. When the cross-sectional area of a wire test piece is too small for this requirement to be met with (L_o)/diameter (d) ratio of 5, a higher value or a non-proportional test piece may be used. Plate test pieces, with a rectangular or square cross-section have a direct relationship between the original gauge length, L_o, and the original cross-sectional area, S_o, expressed by the formula L_o, where k is a coefficient of proportionality, and are called proportional test pieces. The internationally adopted value for k is 5,65. The original gauge length shall be not less than 15 mm. When the cross-sectional area of the test piece is too small for this requirement to be met with, k 5,65, a higher value (preferably 11,3) or a non-proportional test piece may be used (ISO 6892-1:—, 6.1.1). Examples for round bar and plate test pieces are given in Annex A.

7.4 Sub-size test pieces

Special care in fabrication and testing is required for test pieces with diameters less than 6 mm. As the test piece size is reduced, factors such as machining, surface finish, and alignment are of increasing importance.

7.5 Sampling

Remove samples for tensile testing from the material in its final condition to ensure that the properties measured are representative of the product.

Cut test pieces from locations thought to be most representative of the stock material. The conventional locations should normally be used

— for products 40 mm or less in thickness or diameter, the location should be at the centre , and

— for products over 40 mm in thickness or diameter, the location should be midway from the surface to the centre.

8 Testing conditions

8.1 Test piece installation

Install the test piece in the cryostat with sufficient slack in the instrumentation wires so they will not be stretched or crimped during positioning of the dewar or subsequent motions during testing.

During alignment, maintain the applied tensile force below one-third of the proportional limit of the material being tested. Subsequently, maintain a small but sufficient force to ensure that the alignment is retained during cool down.

NOTE A low force control condition is preferable during cooldown to maintain test piece alignment and avoid the application of excessive stress before the start of the tensile test.

8.2 Cooling procedure

Ice can block cryogenic transfer lines or cause erratic loading behaviour if it forms between various parts of the test piece, clip gauge, and force train. To prevent icing, remove any condensate from the apparatus before cooling by drying it thoroughly with an air jet or heat gun. If a clip gauge with a protective casing is used, position the gauge so that cryogenic fluid can enter freely to surround the gauge's active elements to prevent the entrapment of gas bubbles and the associated clip gauge noise.

Next, position the dewar and pre-cool the apparatus by transferring liquid nitrogen into the cryostat. After boiling subsides (an indication that thermal equilibrium is reached), remove all the liquid nitrogen from the cryostat, and transfer liquid helium into the cryostat until the test piece and grips are fully submerged. Testing may begin after the system has reached thermal equilibrium at 4 K. The test piece shall remain fully submerged at all times during the test.

NOTE The heat-transfer characteristics of gaseous helium are lower than those of liquid helium; therefore it is imperative that the test pieces remain submerged in liquid helium to minimize the influence of generated heat on the mechanical property measurements.

8.3 Rate of testing

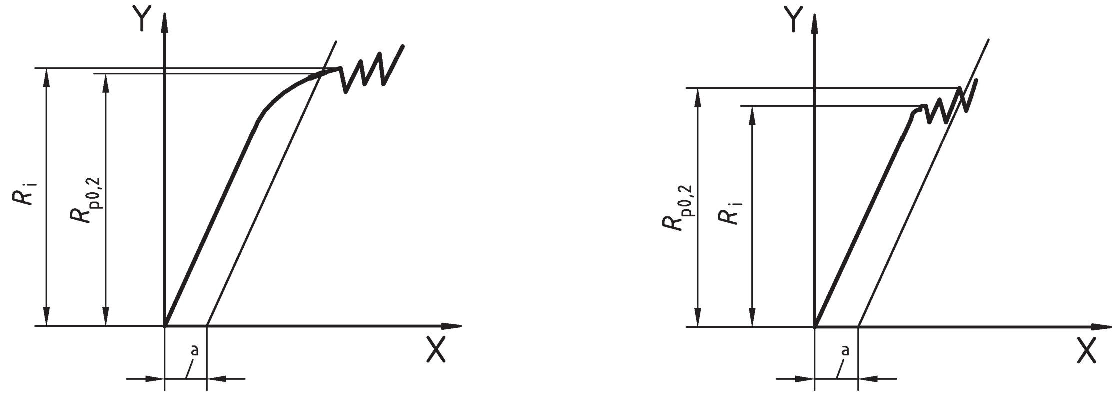
8.3.1 Rate limit

High strain rates may result in excessive test piece heating and therefore are not acceptable for mechanical property measurements of materials. Lower rates may result in extended test time and higher helium consumption.

8.3.2 Rate selection

It is recommended that the crosshead rate be chosen such that the nominal strain rate, based on the length of the reduced section, is 1×10^{-4} s^{-1} with a relative tolerance of ±20 %. However, any convenient crosshead displacement rate may be used to reach an applied stress of one-half the proof strength; after that, the crosshead displacement rate shall be chosen so that the nominal strain rate never exceeds1 × 10^{-3} s^{-1}with a relative tolerance of ±20 %.Strain rates ranging from 10^{-5} s^{-1} to 10^{-3} s^{-1} are often used for tensile tests at 4 K, but some alloys are moderately sensitive to strain rate variations in this range. Some high strength austenitic steels show mild transitions in tensile properties at strain rates in the range 10^{-4} s^{-1} to 10^{-3} s^{-1}, and other alloys with high ratios of strength to thermal conductivity (e.g. titanium alloys) may show similar trends. Consequently, it may be desirable in some tests to use strain rates much lower than the 1×10^{-3} s^{-1} maximum allowed by this part of ISO 6892.

A change of strain rate may be beneficial. For example, the strain required to initiate discontinuous yielding typically increases with decreasing strain rate. If the first serration occurs near 0,2 % plastic strain, it may be possible to reduce the speed of the test to postpone the first serration, and to prevent interference in the measurement of the proof strength (see Figure 3). This may be accomplished by using an initially low strain rate to determine the proof strength followed by an increase of the strain rate to complete the test.



a) Serration after 0,2 % strain

b) Serration before 0,2 % strain

Key

Y stress

X strain

[a] 0,2 % offset.

Figure 3 — Stress-strain diagram for determination of 0,2 % proof strength ($R_{p0,2}$) by the offset method

9 Procedure

9.1 Determination of original cross-sectional area (S_o)

The original cross-sectional area shall be calculated from the measurements of the appropriate dimensions with an error not exceeding ±0,5 % or 0,010 mm, whichever is greater.

9.2 Marking of the original gauge length (L_o)

Gauge marks may be scribed, or inked at appropriate locations on the parallel length of the test piece. After marking the gauge length, measure it to the nearest 0,1 mm.

For metals of low ductility, gauge marks punched or scribed on the parallel length may induce failure at those locations due to stress concentrations. To avoid this, coat the parallel section with layout ink, and mark the gauge length by rotating the test piece in a device with knife edges scraping off the ink at the appropriate intervals. Otherwise, gauge marks may be placed on the test piece shoulders, or the overall length of the test piece may be used to determine elongations; in that case some error is introduced from measurement across section changes and the results should be qualified.

9.3 Determination of percentage elongation after fracture (A)

Percentage elongation after fracture shall be determined in accordance with the definition given in Table 1.

9.4 Determination of the 0,2 % proof-strength, plastic extension ($R_{p0,2}$)

The proof strength ($R_{p0,2}$) is determined by a computer software program or from the force-extension diagram by constructing a line parallel to the linear portion of the curve at a distance equal to the prescribed plastic percentage extension, i.e. 0,2 %. The point at which this line intersects the curve gives the force corresponding to the desired proof strength. The latter is obtained by dividing this force by the original cross-sectional area of the test piece (S_o). If the 0,2 % offset line intersects the curve at a force associated with discontinuous yielding, then the highest stress before decrease is reported conventionally as the proof strength (see Figure 3).

9.5 Discontinuous yielding strength (R_i)

Calculate the stress corresponding to the point of initiation of the first discontinuous-yielding event by dividing the maximum force attained at the beginning of the first measurable serration (see Figure 3) by the cross-sectional area of the test piece.

9.6 Tensile strength (R_m)

Calculate the tensile strength by dividing the maximum force attained by the test piece during the tensile test by the original cross-sectional area of the test piece.

9.7 Reduction of area (Z)

Percentage reduction of area shall be determined in accordance with the definition given in Table 1.

10 Test report

The test report shall include at least following information:

a) a reference to this part of ISO 6892, i.e. ISO 6892-4:2015;

b) material characterization — identification of material, manufacturing, processing, heat treatment condition, and metallurgical information;

c) test piece characterization — the test piece location and its orientation relative to the principal working directions. Also report the test piece dimensions, including the cross-section dimensions, the transition radius, the parallel length;

d) strain rate — the crosshead separation rate and nominal strain rate for the entire test. If the crosshead separation rate was changed during the test, the effective nominal strain rates before and after the rate was changed shall be reported. The strain rates are calculated based on the extensometer gauge length when the new crosshead separation rate is adopted;

e) test results — the 0,2 % proof strength, the tensile strength, the percentage elongation after fracture and the method of its calculation, the gauge length/diameter ratio for cylindrical test pieces, and the percentage reduction of area.

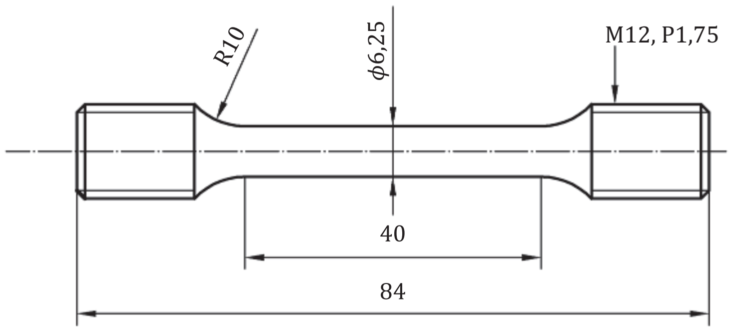
NOTE The following information are optional: Modulus of elasticity at 4 K, slope of the elastic part of the stress-percentage extension curve, the force-extension curve and the force-displacement curve, fracture mode and location of test piece, working condition, the manufacturer's name, the average grain-size of the test material, room temperature mechanical properties, compliance of the testing machine including the cryostats, type and capacity of testing machine, type of cryogenic apparatus, type and performance of extensometer (the available calibration plot), stress-strain curve of the measurement, especially the stress-strain regime between 0 % to 1 %, the discontinuous yielding stress and the strain rate at which it was measured. If replicate test pieces are tested, report the number of tests, the average value of all mechanical property measurements, and a measure of the scatter of the data.

11 Measurement uncertainty

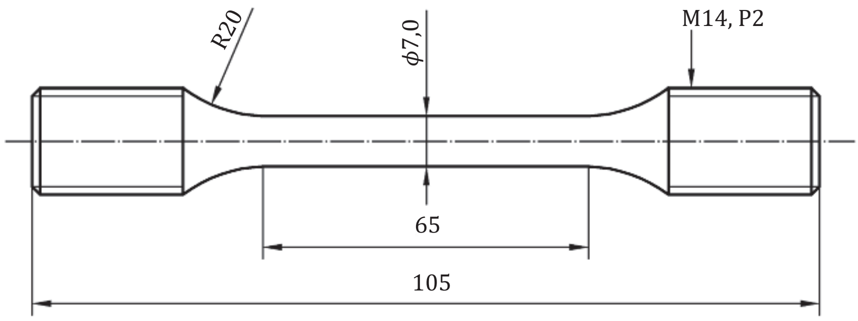
For consideration of uncertainty, see ISO 6892-1:—, Annex K, Estimation of the Uncertainty of Measurements, which provides guidance for the determination of uncertainty related to metrological parameters .

Annex A
(informative)

Examples of test pieces for tensile testing in liquid helium



a)



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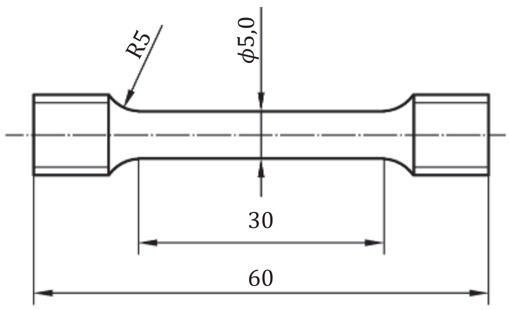

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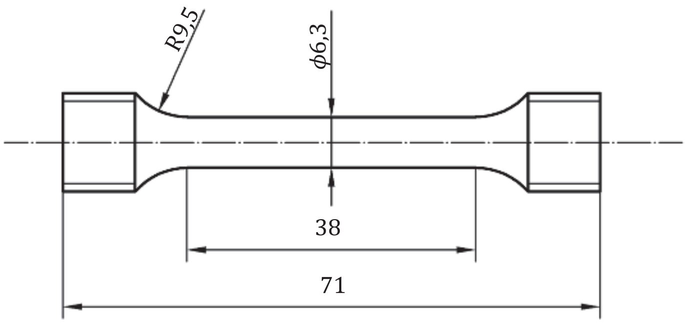

d)

Figure A.1 — Bar test pieces with threated ends

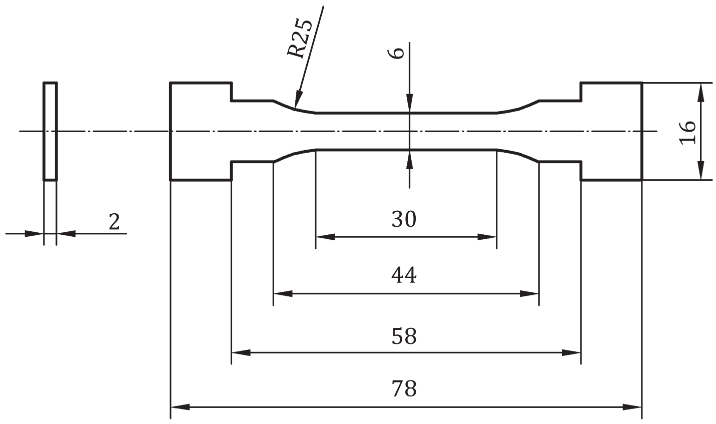

Figure A.2 — Plate test piece with shoulders

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[1] ASTM E 1450, *Standard test method for tension testing of structural alloys in liquid helium*

[2] JIS Z 2277, *Method of tensile testing for metallic materials in liquid helium*

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