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Metallic materials — Tensile testing in liquid helium

Matériaux métalliques — Essai de traction dans l'hélium liquide

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

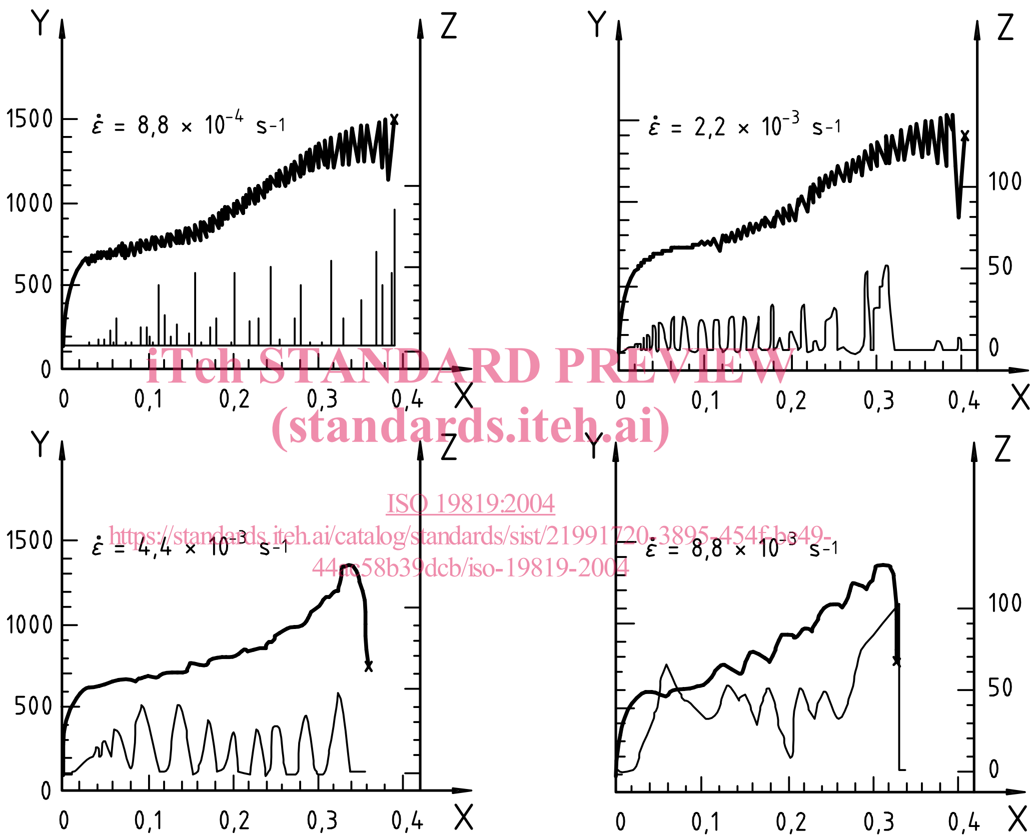
The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 19819 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 1, *Uniaxial testing*.

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 specimen heating. Examples of serrated stress-strain curves for a typical austenitic stainless steel with discontinuous yielding are shown in Figure 1.



Key

X strain (deformation)

Y stress (unit force), N/mm^2

Z temperature, K

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

A constant specimen temperature cannot be maintained at all times during testing in liquid helium. Due to adiabatic heating, the specimen 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 specimen size and test speed. 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 nearly isothermal, and discontinuous yielding typically does not occur.

The stress-strain response of a material tested in liquid helium depends on whether force control or displacement control is used. Displacement control is specified in this International Standard since the goal is material characterization by conventional methods. The possibility of a different and less favourable material response shall be taken into account when data are used for design in actual applications subject to force-controlled conditions.

Metallic materials — Tensile testing in liquid helium

1 Scope

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

This International Standard may also apply to tensile testing at cryogenic temperatures (less than – 196 °C or 77 K), which requires special apparatus, smaller specimens, and concern for serrated yielding, adiabatic heating and strain rate effects.

To conduct a tensile test at 4 K in accordance with this International Standard, the specimen 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. Tests using force control or higher strain rates are not considered.

2 Normative references

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

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

ISO 9513:1999, *Metallic materials — Calibration of extensometers used in uniaxial testing*

ISO 15579, *Metallic materials — Tensile testing at low temperature*

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 15579 and the following apply.

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

3.2
axial strain
average of the longitudinal strains measured at opposite or equally-spaced surface locations on the sides of the longitudinal axis of symmetry of the specimen

NOTE The longitudinal strains are measured using two or more strain-sensing transducers located at the mid-length of the parallel length.

1) To be published. (Revision of ISO 7500-1:1999)

3.3
bending strain
difference between the strain at the surface of the specimen and the axial strain

NOTE The bending strain varies around the circumference and along the parallel length of the specimen.

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 specimens in cryogenic environments

See Figure 2.

4 Symbols and designations

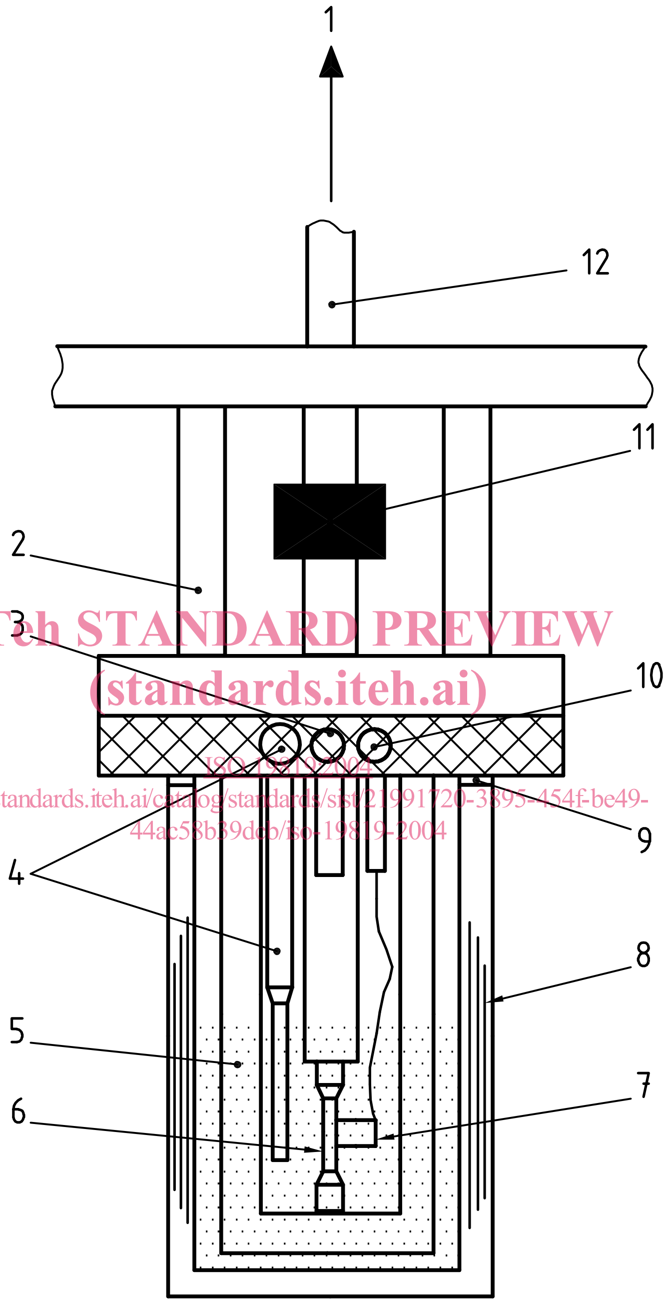
Symbols and corresponding designations are given in Table 1.

Table 1 — Symbols and designations

Symbol	Unit	Designation
A	%	Percentage elongation after fracture: $A = \frac{L_u - L_o}{L_o} \times 100$
d	mm	Diameter of the parallel length of a cylindrical test piece or diameter of a circular wire
F_m	N	Maximum force
L_c	mm	Parallel length
L_e	mm	Extensometer gauge length
L_o	mm	Original gauge length
L_u	mm	Final gauge length after fracture
R_i	N/mm^2	Discontinuous yielding strength
R_m	N/mm^2	Tensile strength
$R_{p0,2}$	N/mm^2	0,2 % proof strength, non-proportional extension
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: $Z = \frac{S_o - S_u}{S_o} \times 100$

5 Principle

Using a tensile force, the test consists of straining a specimen in liquid helium, generally to fracture, for the purpose of determining one or more of the mechanical properties defined in Clause 3.



Key

1 force

2 room temperature load frame

3 vent

4 vacuum-insulated transfer tube

5 cryogenic load frame

6 specimen

7 extensometer

8 vacuum-insulated dewar

9 dewar seal

10 electrical feed-through

11 load cell

12 pull rod

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

6 Apparatus

6.1 Testing machine

6.1.1 General

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.

6.1.2 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 specimen or by using a special calibration specimen. Then measure the compliance at a low force and at the highest force used to qualify the machine, as indicated in 6.1.5.

NOTE Different compliances may alter the elongation and tensile strength of materials, because larger discontinuous deformation occurs in a lower compliance test facility.

6.1.3 System design

Typically, alloys in liquid helium exhibit double or triple their strengths at ambient temperature. For the same specimen geometry, higher forces shall be applied to the cryostat, specimen, 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 specimens described in 7.2.

6.1.4 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 in order 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 (e.g., glass-epoxy composites) are excellent insulators and are sometimes used for compression members.

6.1.5 Alignment

Proper system alignment is essential in order to minimize bending strains during the tensile testing. The machine and grips should be capable of applying force to a precisely machined calibration specimen so that the maximum bending strain does not exceed 10 % of the axial strain. Reduce bending strain to an acceptable level by making proportional adjustments to a cryostat that has alignment capability, or by using spacing shims to compensate an unadjustable fixture. Calculate the strain based on readings taken while the calibration specimen 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 on the apparatus, the specimen form and cryostat should be the same as that used during cryogenic tests, and the specimen concentricity should be as near perfect as possible. No plastic strain should occur in the parallel length of the alignment specimen during loading. In some cases this may necessitate the use of a relatively stiff, high-strength calibration specimen.

For cylindrical specimens, calculate the maximum bending strain, defined in 3.3, from the strain measured with three electrical resistance strain gauges, extensometers or clip gauges placed at circumferential positions, equally-spaced around and at the centre of the parallel length of the specimen.

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

With a strain-averaging technique, nonaxiality of loading (which may be introduced due to the machining of the specimens) is usually sufficient to introduce errors in tensile tests at small strains when strain is measured at only one position on the specimen. Therefore measure strains at three equally-spaced (or, if good alignment has been achieved, at least two opposing) positions within the parallel length. Report the average of the strains from the two or three positions centred on the parallel length.

6.1.6 Gripping mechanisms

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

6.2 Cryostats and support apparatus

6.2.1 Cryostats

A cryostat capable of retaining liquid helium is required. In general, cryostat load frames for existing test machines shall be custom-built, but they may accommodate commercially available dewars. The cryostat may possess adjustable load columns to facilitate alignment.

6.2.2 Dewars

Stainless steel dewars are safer (that is, more fracture resistant) than glass ones. 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-jacket 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 specimen completely immersed, a thermocouple to measure its temperature is not required for routine tests. Instead of that, a simple indicator or meter is required to ensure that the specimen 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 specimen. 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 9513:1999) for the determination of the 0,2 % proof strength; for the determination of other properties (corresponding to higher elongations) extensometers of class 2 (see ISO 9513:1999) can be used.