INTERNATIONAL STANDARD

ISO
22674

Dentistry — Metallic materials for fixed and removable restorations and appliances

Médecine bucco-dentaire — Matériaux métalliques pour les restaurations fixes et amovibles et les appareils

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Contents

Page

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.

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

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

This document was prepared by Technical Committee ISO/TC 106, *Dentistry,* Subcommittee SC 2, *Prosthodontic materials],* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 55, *Dentistry,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 22674:2016), which has been technically revised.

The main changes are as follows:

— addition of products produced using additive and subtractive manufacturing;

— revision of definitions and addition of new definitions for modern manufacturing techniques;

— addition of an overview of symbols in Clause 4 as Table 1;

— harmonization of symbols in formulae and Figures;

— static determination of elastic modulus was added in 8.4.1.3 (as an additional option);

— a requirement for a test report was added as Clause 9;

— a requirement for labelling the alloy composition on the package was added in 10.4.

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

Introduction

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this document, but it is recommended that, in assessing possible biological hazards, reference be made to ISO 10993-1 and ISO 7405.

Requirements for the performance of metals and alloys used for the metallic component of a metal-ceramic restoration contained in this document supersede such requirements formerly contained in ISO 9693. The requirements for the performance of ceramic material and the metal-ceramic bond in metal-ceramic restorative systems are specified in ISO 9693.

Requirements for the proof stress and minimum elongation after fracture for Type 0 metallic materials are not included in this document, but it is recommended to adopt the test procedure given in Annex A when measuring these properties.

Dentistry — Metallic materials for fixed and removable restorations and appliances

1 Scope

This document specifies requirements and test methods for metallic materials that are suitable for the fabrication of dental restorations and appliances. Included are metallic materials recommended for use either with or without a ceramic veneer, or recommended for both uses. Furthermore, this document specifies requirements for packaging and marking of the products and for the instructions for use of these materials, including products delivered for sale to a third party.

This document does not apply to alloys for dental amalgam (see ISO 24234), dental brazing materials (see ISO 9333), or metallic materials for orthodontic appliances (e.g. wires, brackets, bands and screws).

This document is not applicable to magnetic attachment, which are specified in ISO 13017.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1942, *Dentistry — Vocabulary*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5832-2, *Implants for surgery — Metallic materials — Part 2: Unalloyed titanium*

ISO 5832-3, *Implants for surgery — Metallic materials — Part 3: Wrought titanium 6-aluminium 4-vanadium alloy*

ISO 6344-3, *Coated abrasives — Determination and designation of grain size distribution — Part 3: Microgrit sizes P240 to P5000*

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

ISO 7500-1, *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*

ISO 9693, *Dentistry — Compatibility testing for metal-ceramic and ceramic-ceramic systems*

ISO 10271:2020, *Dentistry — Corrosion test methods for metallic materials*

ISO 15223-1:2021, *Medical devices — Symbols to be used with information to be supplied by the manufacturer — Part 1: General requirements*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following apply.

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

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

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

3.1
additive manufacturing
process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to *subtractive manufacturing* (3.19) and formative manufacturing methodologies

[SOURCE: ISO/ASTM 52900:2021, 3.1.2, modified — Notes 1 and 2 to entry have been deleted.]

3.2
appliance
prefabricated metallic device such as attachments and bars

3.3
as-cast state
metallurgical condition of the *metallic material* (3.15) in its solid state when removed from the casting machine

Note 1 to entry: This condition is dependent upon the manufacturer's recommended cooling procedure (e.g. bench cooling).

3.4
base metal
metallic element, with the exception of *noble metals* (3.16) and silver

3.5
base-metal alloy
alloy having a *base metal* (3.4) as the principal element

3.6
bench-cooling
process whereby a casting is retained in its investment with exposed metal uppermost and placed on a flat, insulating surface at ambient temperature in freely circulating air until its temperature falls to ambient

3.7
casting alloy
metallic material (3.15) designed for casting into an investment mould

3.8
ceramic veneer
thin ceramic surface layer present on a *metallic material* (3.15) restoration to provide an aesthetic effect

3.9
elastic modulus
Young's modulus
ratio of elastic stress to elastic strain

3.10
hardening
heat-treatment of a *metallic material* (3.15) producing a condition which provides a higher 0,2 % proof stress than the *as-cast state* (3.3)

Note 1 to entry: If recommended by the manufacturer, explicit instructions are required in the instructions for use.

3.11
hazardous element
element that is known for its potential to produce an adverse biological effect

Note 1 to entry: The presence of such an element (as an alloying addition or as an impurity) in a dental alloy does not imply that the alloy, in itself, is hazardous.

3.12
heat treatment
thermal treatment of a *metallic material* ([3.15](#)) such as a material stress relieving process, including *softening* ([3.18](#)), *hardening* ([3.10](#)), ceramic firing

3.13
metal-ceramic restoration
dental restoration with a *ceramic veneer* ([3.8](#)) bonded to a *metallic material* ([3.15](#)) substructure by firing

Note 1 to entry: This can apply also to the metallic material used for such a restoration. In this context, metal-ceramic alloy is a synonym.

Note 2 to entry: If recommended, such a metallic material may be used without a ceramic veneer.

3.14
metallic base
metal with highest concentration by mass fraction in the alloy

3.15
metallic material
material with properties that are associated with an alloy, *noble metal* ([3.16](#)) or *base metal* ([3.4](#))

Note 1 to entry: This may be a pure element, commercially pure metal or an alloy.

3.16
noble metal
metal containing gold, metal of the platinum group and/or rhenium

3.17
one-surface inlay
inlay restoration that is exposed to the oral environment on one and no more of the surfaces that are used to define the tooth for the purposes of charting

3.18
softening
heat-treatment of a *metallic material* ([3.15](#)) producing a condition which provides a lower 0,2 % proof stress than the *as-cast state* ([3.3](#))

Note 1 to entry: If recommended by the manufacturer, explicit instructions are required in the instructions for use.

3.19
subtractive manufacturing
manufacturing process where a solid material such as milling blank is reduced by milling, grinding, eroding or similar techniques to the final shape

4   Symbols and classification

4.1   Symbols

[Table 1](#) gives an overview about the symbols used in this document.

Table 1 — Symbols and their usage

Symbol	Description	Usage
$A_{15\ mm}$	percentage elongation after fracture	A.3.3, A.3.4
b	breadth	8.4.2.2, A.1.1, Clause B.2, B.2.1.1.1, B.4.1, C.3.2.1, C.3.2.2, Figures 3 and A.1, Formulae (3), (4), (5), (B.2), (B.5), (B.6) and (B.7), Tables 4 and 5
C_1	correction factor 1 for the elastic modulus	8.5.2, Formulae (2) and (3)
C_2	correction factor 2 for the shear modulus	8.5.3, Formulae (4) and (5)
D	minimum difference between two readings that the measuring instrument can discriminate	B.1, Formulae (B.1)
d	deflection	8.4.2.2, Formulae (3) and (6), Table 4
E	elastic modulus	B.2.1.2, B.3.1, B.4.2, Formulae (1), (3), (6), (B.1), (B.4), (B.5), (B.6), (B.7) and (C.1), Table 4
E_a	apparent elastic modulus	8.4.2.2, 8.4.2.3, C.3.2.2, C.3.2.3, Formula (C.1), Table 4
F	force	8.4.2.2, Table 4
F_5	force required to bring the lowest stressed part of the specimen to 5 % of material's $R_{P0,2}$	8.4.2.2
F_{60}	force required to bring the most highly stressed part of the specimen to 60 % of material's $R_{P0,2}$	8.4.2.2, Table 4
f_1	fundamental frequency measured in the flexural mode of vibration from acoustic measurement	8.5.2, B.4.2, B.4.3, Formulae (3), (B.7) and (B.8)
f_2	fundamental frequency measured for the torsional mode of vibration	8.5.3, B.4.2, B.4.3, Formulae (5), (B.7) and (B.8)
G	shear modulus	Formulae (5) and (6)
h	thickness (height)	8.4.2.2, 8.5.2, A.1.1, C.3.2.2, B.2.1.1.1, B.4.1, B.4.3, Figures 3 and A.1, Formulae (2), (3), (4), (5), (B.2), (B.5), (B.6), (B.7) and (B.8), Tables 4 and 5
h_1	greatest measured thickness	7.5.3, 7.5.4, Figure 4
h_2	least measured thickness	7.5.3, 7.5.4, Figure 4
l	length of specimen	8.5.2, B.4.1, B.4.2, B.4.3, A.1.1, Clause B.2, Figures 3, 6 and 7, Formulae (2), (3), (5), (B.7) and (B.8)
l_f	free length between grips	A.1.1, Figure A.1
l_g	initial gauge length	7.3, 8.4.1.2, A.3.4, Figures 1, 2 and A.1, Table 4
l_p	parallel length	7.3, Figures 1 and 2, Table 4
L_i	inner separation of load rollers	8.4.2.1.3, B.3.1, Figure 5, Formula (B.6), Table 4
L_o	outer separation of support load rollers	8.4.2.1.3, B.3.1, Figure 5, Formula (B.6), Table 4
m	mass	8.4.3.2, 8.5.2, B.4.1, Formulae (3), (5), (B.7)

Table 1 *(continued)*

Symbol	Description	Usage
n	number of individual readings	B.1, Formula (B.1)
q	mean of n individual readings	B.1, Formula (B.1)
q_i	value of the ith series of readings	B.1, Formula (B.1)
r	radius of specimen	B.2.1.1.2, Formula (B.3)
$R_{P0,2}$	proof stress of 0,2 % non-proportional extension	8.3.3.1, 8.4.2.2, A.3.1.3, A.3.3, A.3.4, Tables 3 and 4
S	cross-sectional area	7.5.2, 8.4.1.2, B.2.1.1.1, B.2.1.1.2, B.2.1.2, Formulae (1), (B.2), (B.3) and (B.4)
S_0	original cross-sectional area	A.3.3, A.3.4
$u(S)$	standard uncertainty in the measurement of cross-section	B.2.1.1.1, B.2.1.1.2, B.2.1.2, Formulae (B.2), (B.3) and (B.4)
$u(b)$	standard uncertainty in the measurement of breadth of specimen	B.2.1.1.1, B.3.1, Formulae (B.2), (B.5), (B.6) and (B.7)
$u(d)$	standard uncertainty in the measurement of Δd	B.3.1, Formulae (B.5) and (B.6)
$u(E)$	standard uncertainty in the measurement of elastic modulus	B.2.1.2, B.3.1, B.4.2, C.3.2.2, C.3.2.3, Formulae (B.4), (B.5), (B.6), (B.7), (B.8) and (C.1)
$u(F)$	standard uncertainty in the measurement of load force	B.2.1.2, B.3.1, Formulae (B.4), (B.5) and (B.6)
$u(f)$	standard uncertainty in frequency	B.4.1, B.4.2, B.4.3, Formulae (B.7) and (B.8)
$u(h)$	standard uncertainty in the measurement of thickness of specimen	B.2.1.1.1, B.3.1, Formulae (B.2), (B.5), (B.6), (B.7) and (B.8)
$u(l)$	standard uncertainty in the measurement of length of specimen	Formulae (B.7) and (B.8)
$u(L_o)$	standard uncertainty in the measurement of L_o	B.3.1, Formula (B.5)
$u(L_i)$	standard uncertainty in the measurement of L_i	B.3.1
$u(m)$	standard uncertainty in the measurement of mass	Formula (B.7)
$u(q)$	standard uncertainty in the measurement of q	B.1, Formula (B.1)
$u(r)$	standard uncertainty in the measurement of the radius of the specimen	B.2.1.1.2, Formula (B.3)
$u(v)$	combined standard uncertainty in Poisson's ratio	B.4.3, Formula (B.8)
$u(\Delta L)$	standard uncertainty in the measurements of ΔL	B.2.1.2, Formula (B.4)
v	Poisson's ratio	8.4.2.2, 8.5.1, B.4.3, C.3.2.3, Formulae (2), (6), (B.8), (C.1) and (C.2)
Δd	change in displacement of mid-point of specimen corresponding to the change ΔF in load force	B.3.1, Formulae (B.5) and (B.6)
ΔF	change in load force corresponding to the change in gauge length	8.4.1.2, B.2.1.2, B.3.1, Formulae (1), (B.4), (B.5) and (B.6)
Δh	separation of outer and inner reference planes	7.5.3, 7.5.4, Figure 4
ΔL	change in gauge length measured by the extensometer	8.4.1.2, B.2.1.2, Formulae (1) and (B.4)

4.2 Classification

For the purposes of this document, a metallic material is classified according to its mechanical properties by a Type number, of which there are six.

Examples of the applications for which these Types are intended are as follows:

— Type 0: intended for low stress bearing single-tooth fixed prostheses, for example, small veneered one-surface inlays, veneered crowns;

NOTE 1   Metallic materials for metal-ceramic crowns produced by electroforming or sintering belong to Type 0.

— Type 1: for low stress bearing single-tooth fixed prostheses, for example, veneered or unveneered one-surface inlays, veneered crowns;

— Type 2: for single tooth fixed prostheses, for example, crowns or inlays without restriction on the number of surfaces;

— Type 3: for multiple unit fixed prostheses;

— Type 4: for appliances with thin sections that are subject to very high forces, for example, removable partial dentures, clasps, thin veneered single crowns, full arch fixed dental prostheses or those with small cross-sections, bars, attachments, implant retained superstructures;

— Type 5: for appliances in which parts require the combination of high stiffness and proof stress, for example, thin removable partial dentures, parts with thin cross-sections, clasps.

NOTE 2   The higher application type can include lower application types.

NOTE 3   Multiple-unit and full-arch, fixed dental prostheses are also referred to as bridges.

5   Requirements

5.1   Chemical composition

5.1.1   Reported composition

For all elements that are present in excess of mass fraction of 1,0 %, each constituent element shall be declared by the manufacturer and shall be reported [see 10.1, a)] to a precision of a mass fraction of 0,1 %.

Any element that is present in excess of mass fraction of 0,1 %, and with a lower or equal mass fraction of 1,0 %, shall be identified [see 10.1 a)] either by name or symbol.

If the metallic material contains less than or equal to a mass fraction of 0,1 % of a specified element (other than one named in 5.2), it may be named as "free of" this specified element [see 10.1 p) and 10.4 j)].

If applicable, the name of the metallic base shall precede the words "-based metallic material for dental restoration" or "-based casting alloy" or "-based metal-ceramic material", as is appropriate.

5.1.2   Permitted deviation from the reported composition for elements

The permitted deviation of the reported composition for elements from the value stated on the package or label or insert [see 10.1 a)] is given in Table 2.

Table 2 — Permitted deviation from the reported composition for elements

Alloy	Elemental content	
	1,0 % < w ≤ 20 %	w > 20 %
Base-metal alloy	maximum 1,0 %	maximum 2,0 %
Silver-based and noble metal alloy	maximum 0,5 %	maximum 0,5 %
Key w : mass fraction		

5.2 Hazardous elements

5.2.1 Hazardous elements

For the purposes of this document, the elements nickel, cadmium, beryllium and lead are designated to be hazardous elements.

5.2.2 Limits for the hazardous elements

The metallic material shall not contain more than a mass fraction of 0,02 % of beryllium, cadmium or lead.

Beryllium, cadmium or lead are neither alloying elements nor elements inherent to the manufacturing process of titanium metallic materials. Apply the requirements of chemical composition of ISO 5832-2 and ISO 5832-3 to titanium metallic materials; beryllium, cadmium or lead do not need to be analysed.

5.2.3 Nickel

5.2.3.1 Manufacturer's reported nickel content and permitted deviation

If the metallic material contains more than a mass fraction of 0,1 % of nickel, this content shall be given to an accuracy of mass fraction of 0,1 % in the literature which accompanies the package [see 10.1 n)] and on the package, label or insert [see 10.4 h)].

The mass fraction shall not exceed the value stated in 10.1 n) and 10.4 h).

5.2.3.2 Nickel-free products

For the purposes of this document, alloys with a maximum of mass fraction of 0,1 % nickel can be labelled "nickel free" [see 10.1 o) and 10.4 j)].

If nickel is not declared, it shall be limited to a maximum of 0,1 %. This limit shall be adhered to when nickel is a natural impurity in a component of the alloy.

5.3 Biocompatibility

Refer to the introduction for guidance on biocompatibility.

5.4 Mechanical properties

5.4.1 General

The mechanical properties (see Table 3) shall be met by the metallic material after the recommended processing techniques (e.g. casting, bench-cooling, machining, thermal treatment) and after the ceramic firing schedule (if appropriate) have been applied. A metallic material recommended for use either with or without a ceramic veneer shall meet this requirement in both metallurgical conditions.

If a heat-treatment is recommended by the manufacturer [see 10.2 c)], this requirement shall be met by the material in the heat-treated condition, applied in accordance with the instructions for use.

For a metallic material, the Type can be classified differently (according to Table 3) if it is recommended for use in more than one metallurgical condition (produced by alternative processing schedules).

For every recommended metallurgic state (e.g. ceramic firings or other thermal treatments), the applicable Type shall be specified separately.

Table 3 — Mechanical properties

Type	Minimum proof stress of 0,2 % non-proportional extension $R_{p0,2min}$ MPa	Minimum elongation after fracture %	Minimum elastic modulus GPa
0	—	—	—
1	80	18	—
2	180	10	—
3	270	5	—
4	360	2	—
5	500	2	150

Testing shall be done according to 8.3.

5.4.2 Proof stress of 0,2 % non-proportional extension

5.4.2.1 Determination of compliance

Refer to the classification Type stated in 10.1 b).

If four, five or six of the results for the set of six specimens in the first test series meet the requirement for proof stress of 0,2 % non-proportional extension, the metallic material complies with the requirement.

If two or fewer of the results for the set of six specimens in the first test series meet the requirement for proof stress of 0,2 % non-proportional extension, the metallic material fails to comply with the requirement.

NOTE 1   Two lots of six specimens are produced. One of these lots is tested in the first test series. If required, replacement specimens are drawn from the second lot and used in the first test series. The remaining specimens in the second lot form the second test series.

NOTE 2   In this context, the number six is reached to complete the set in the first test series when the number of specimens tested less those rejected after post fracture examination is six, (i.e. replacement specimens are included in the total).

If three or four specimens in the first lot of six specimens are rejected on the basis of 8.3.2 and replaced with specimens from the second lot, all 12 specimens shall be tested. If at least eight results meet the requirement for proof stress of 0,2 % of non-proportional extension, the metallic material complies with the requirement.

If three of the results for the set of six specimens in the first test series meet the requirement for proof stress of 0,2 % of non-proportional extension, all remaining specimens shall be tested in a second test series. If five or six of the results for the specimens in the second test series meet the requirement for proof stress of 0,2 % of non-proportional extension, the metallic material complies with the requirement.

Under this compliance criterion, if one specimen from the first lot has been rejected on the basis of inspection after fracture (see 8.3.2) during the first test series and a specimen from the second lot

of six used as its replacement, then all five remaining specimens in the second series shall meet the requirement. Only one replacement is possible for a borderline metallic material.

If three of the results for the first set of six specimens in the first test series meet the requirement for proof stress of 0,2 % of non-proportional extension and four or fewer of the results from the specimens in the second test series meet the requirement for proof stress of 0,2 % of non-proportional extension, the metallic material fails to comply with the requirement.

Testing shall be done according to 8.3.2.

5.4.2.2 Mean value

The mean value for the proof stress of 0,2 % non-proportional extension, calculated according to 8.3.3.2 shall not differ by more than 10 % from the value given in the literature accompanying the package [see 10.1 c)] and not be less than the minimum value of the given Type.

5.4.3 Elongation after fracture

5.4.3.1 Determination of compliance with the requirement

Refer to the classification Type stated in 10.1 b).

If the four, five or six of the results for the set of six specimens in the first test series that meet the requirement for proof stress of 0,2 % of non-proportional extension also meet the requirement for elongation after fracture, the metallic material complies with the requirement for elongation after fracture.

If two or fewer of the results for the set of six specimens in the first test series meet the requirement for elongation after fracture, the metallic material fails to comply with the requirement for elongation after fracture.

NOTE 1   Two lots of six specimens are produced (see Clause 7). One of these lots is tested in the first test series. If required, replacement specimens are drawn from the second lot and used in the first test series. The remaining specimens in the second lot form the second test series.

NOTE 2   In this context, the number six is reached to complete the set in the first test series when the number of specimens tested less those rejected after post fracture examination (see 8.3.2) is six, (i.e. replacement specimens are included in the total).

If three or four specimens in the first lot of six specimens are rejected on the basis of 8.3.2 and replaced with specimens from the second lot, all 12 specimens shall be tested. If at least eight results of the specimens that meet the requirement for proof stress of 0,2 % of non-proportional extension also meet the requirement for elongation after fracture, the metallic material complies with the requirement for elongation after fracture.

If three of the results for the first set of six specimens in the first test series that meet the requirement for proof stress of 0,2 % of non-proportional extension also have an elongation after fracture that meets the requirement for elongation after fracture, all remaining specimens shall be tested in a second test series. If five or six of the results for the specimens in the second test series have both an elongation after fracture and a proof stress of 0,2 % of non-proportional extension that meet both requirements, the metallic material complies with the requirement for the percentage elongation after fracture. If the results for the five or six specimens in the second series meet the requirement for elongation after fracture, but not the requirement for proof stress of 0,2 % of non-proportional extension, the metallic material fails to comply with requirement for elongation after fracture.

Under this compliance criterion, if one specimen in the first lot has been rejected on the basis of inspection after fracture (8.3.2) during the first test series and a specimen from the second lot of six used as its replacement, then all five remaining specimens in the second series shall meet the requirement. Only one replacement is possible for a borderline metallic material.

If three of the results for the first set of six specimens in the first test series meet the requirement for the percentage elongation after fracture and four or fewer of the results from specimens in the second test series meet the requirement for elongation after fracture, the metallic material fails to comply with the requirement for elongation after fracture.

5.4.3.2   Mean value

The mean value of percentage elongation after fracture calculated according to 8.3.4.2 shall exceed 70 % of the value stated in the literature accompanying the package and shall not be less than the minimum value for the Type [see 10.1 d)] and not be less than the minimum value of the given Type.

5.5   Elastic modulus

5.5.1   Precision of measurement method

The measurement of elastic modulus shall be performed by a method (see 8.4) capable of a precision that gives a combined standard uncertainty less than 15 % of the measured value.

NOTE 1   For guidance on the determination of combined standard uncertainty, see ISO/IEC Guide 98-3.

NOTE 2   For a metallic material with elastic modulus greater than 150 GPa, the method in 7.5.2 does not achieve a precision of 10 %.

5.5.2   Determination of compliance with the requirements for Type 5 materials

Refer to classification Type stated in 10.1 b).

Where compliance with Type 5 is claimed, the mean value of the elastic modulus as determined according to 8.4 shall be at least 150 GPa.

5.5.3   Mean value

The mean value of the elastic modulus shall not differ from the value stated in the literature accompanying the package [see 10.1 e)] by more than 10 % and not be less than the minimum value of the given Type.

5.6   Density

When tested in accordance with 8.8, the density of the metallic material shall not differ by more than ±5 % of the value stated in the literature accompanying the package [see 10.1 f)].

5.7   Corrosion resistance for material integrity

When tested in accordance with 8.7, the total metal ion release from the metallic material into the specified solution (see 8.7.4) at (37 ± 1) °C in a time period of 7 d ± 1 h shall not exceed 200 µg·cm^{-2}.

This requirement shall be met by the metallic material after the recommended processing techniques (e.g. casting, bench-cooling, machining) and after the ceramic firing schedule (if appropriate) have been applied. A metallic material recommended for use either with or without a ceramic veneer shall meet this requirement in both metallurgical conditions.

5.8   Tarnish resistance

This requirement shall apply only if a claim is made in the "Information" of the literature that accompanies the package that the product is "tarnish–resistant".

When tested in accordance with 8.8 or 8.9, if there is no more than a very minor colour change and the products of tarnish are easy to remove by gentle rubbing or brushing, the alloy may be described in the literature accompanying the package as "tarnish-resistant" [see 10.1 g)].

This requirement shall be met by the metallic material after the recommended processing techniques (e.g. casting, bench-cooling, machining) and after the ceramic firing schedule (if appropriate) have been applied. A metallic material recommended for use either with or without a ceramic veneer shall meet this requirement in both metallurgical conditions.

5.9 Solidus and liquidus temperatures (alloy) or melting point (commercially pure metal)

For an alloy with a solidus temperature below or equal to 1 200 °C, the solidus and liquidus temperatures shall not differ by more than ±20 °C from the value stated on the literature accompanying the package [see 10.1 h)] when tested in accordance with 8.10.

For an alloy with a solidus temperature above 1 200 °C, the solidus and liquidus temperatures shall not differ by more than ± 50 °C from the value stated on the literature accompanying the package [see 10.1 h)] when tested in accordance with 8.10.

For a commercially pure metal, the melting point shall not differ from the value stated on the literature accompanying the package [see 10.1 h)] by more than ±20 °C, if it is below or equal to 1 200 °C and by more than ±50 °C if it is above 1 200 °C when tested in accordance with 8.10.

5.10 Thermal expansion coefficient

This requirement shall apply only to a metallic material intended for use in a metal-ceramic restoration.

When tested in accordance with 8.11, the coefficient of linear expansion of the metallic material shall not differ by more than $(0,5 \times 10^{-6})$ K^{-1} from the value stated on the literature accompanying the package [see 10.1 i)].

5.11 Information, instructions and marking

All the requirements specified in Clause 10 shall be present and correct. If detailed instructions or recommendations are required, these shall be adequate to fulfil their purpose.

6 Sampling

The sample shall be adequate to prepare the specimens required in Clause 7 and shall be from the same lot of material. Further samples and packaging materials shall be made available for inspection in accordance with 8.1.

When metallic materials for shaping by numerically controlled methods are supplied in a form unsuitable for a given test, specimens suitable for measuring mechanical properties (see 7.3) may be prepared from bulk metallic material using the same production lot and subject to the same thermo-mechanical treatment.

7 Preparation of specimen

7.1 General

Prepare all specimen following the instructions for use, as detailed in 10.2.

If the specimen are cast, carefully separate the sprues and remove any casting beads, fins, etc.

Additive manufacturing of the test specimen is possible, if this procedure is intended and described by the manufacturer in the instructions for use.

Replace any specimen that have visible defects.

7.2 Heat treatment

7.2.1 General conditions

Test the metallic material against the requirements when it is in the metallurgical condition(s) appropriate to its intended application(s).

7.2.2 Metallic materials for which a heat treatment is recommended in the instructions for use

If a heat-treatment is recommended by the manufacturer [see 10.2 c)], perform the tests in the heat-treated condition in accordance with the instructions for use.

7.2.3 Metallic material for metal-ceramic restorations

Test after the following simulated ceramic-firing schedule has been applied: simulate the oxidation procedure and four ceramic firings at the highest temperature allowed for ceramics recommended for fusing to the metallic material [in accordance with 10.1 k)]. Remove and place the specimens on a ceramic plate (which is at room temperature) to cool to room temperature after the oxidation and ceramic firing simulation.

If the product is recommended also for use without application of a ceramic veneer, also test with the metallic material in the condition given in 7.2.2 or 7.2.4, as appropriate.

7.2.4 Metallic materials for which no heat-treatment is recommended in the instructions for use

Test in the processed condition.

For metallic materials intended for casting, the appropriate condition is the bench-cooled state.

7.3 Proof stress of 0,2 % non-proportional extension and the elongation after fracture: Metallic materials for which production of conventional specimens is possible

The specimen for the tensile test shall have the following dimensions: the gauge length l_g shall be (15 ± 0,1) mm. The parallel section of the specimen l_p shall be (18 ± 0,1) mm.

Prepare two lots of six specimens that comply with Figure 1 or Figure 2, formed, finished and heat-treated in accordance with 7.1 and 7.2.

If the product is intended for use both with and without a ceramic veneer, four lots of six specimens are required, two treated in accordance with 7.2.3 and the two others to be treated in accordance with 7.2.2 or 7.2.4 (as applicable).

Specimen normally require no further finishing after the treatment described in 7.1 and 7.2. If, however, cast specimens are machined instead of polished according to the instructions for use [see 10.2 a)], record this.

Add fine marks or scribe lines to define the ends of the gauge length.
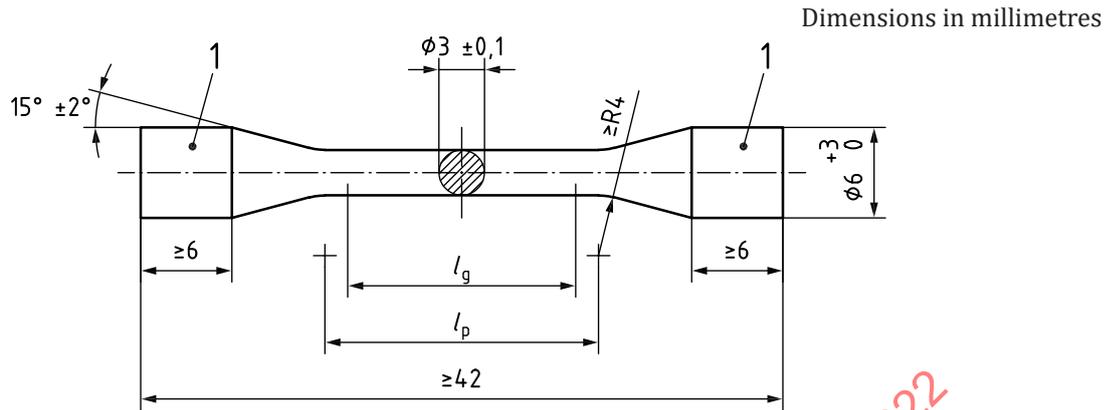
Dimensions in millimetres

Key

1   gripped portion

l_g initial gauge length of (15 ± 0,1) mm

l_p parallel section of (18 ± 0,1) mm

Figure 1 — Specimen with conical shoulders
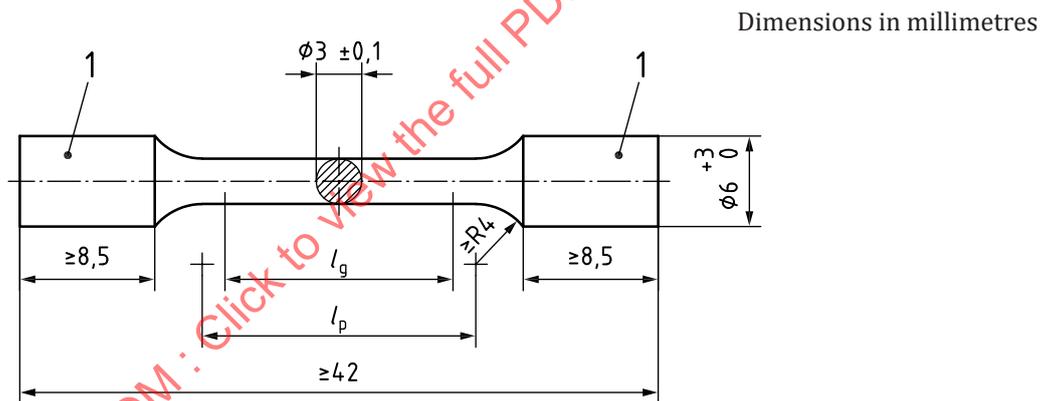
Dimensions in millimetres

Key

1   gripped portion

l_g initial gauge length of (15 ± 0,1) mm

l_p parallel section of (18 ± 0,1) mm

Figure 2 — Specimen with radial shoulders

7.4   Type 0 metallic materials for which the production of conventional specimens is not possible

See Annex A for guidance on metallic materials or semi-finished products for which the processing techniques do not allow the formation of specimen with the shape and dimensions shown in Figure 1 or Figure 2.

7.5   Elastic moduli

7.5.1   General

Three alternative methods of measurement are recommended.

Prior to measurement, the precision of a method can be estimated according to Annex B from the calibrated characteristics of the instruments employed and the approximate elastic modulus of the metallic material.

If the anisotropic elastic properties of the metallic material need to be qualified, the tensile strain method or flexure method shall be used. Prepare two sets of three specimens with the longest dimension parallel and normal to the direction of processing, respectively. Alternatively, the acoustic resonance method may be employed.

7.5.2 Tensile strain method

For the tensile strain method for the measurement of the elastic modulus, prepare three specimens according to 7.3. Provided the average cross-sectional area, S, of the gauge length can be determined to a precision for which the standard uncertainty in S is less than 2 % of the measured value, these specimens can be used directly.

Where estimation of the measurement precision shows that the requirement of 5.5.1 may not be met, specimens with longer gauge length and greater diameter may be substituted. For such specimens, the ratio of gauge length to diameter shall be 4,7 ± 0,3.

Where, however, specimens nominally conforming to 7.3 are found to have an irregular cross-section that cannot be determined with sufficient precision, it is recommended that they be machined to a uniform cross-section over the parallel section and finished with P1200 wet silicon carbide paper complying with ISO 6344-3 before treatment corresponding to the instructions for preparation for ceramic veneering, if this is the intended application.

NOTE   Specimens previously employed for repeated measurements of elastic modulus will subsequently give increased values of yield stress compared to those that have not.

7.5.3 Flexure method (three or four point bending)

For the flexure method, prepare three specimens with dimensions of approximately 34 mm × 13 mm × 1,5 mm and grind, finishing with P1200 wet silicon carbide paper complying with ISO 6344-3, to obtain dimensions that comply with Figure 3.
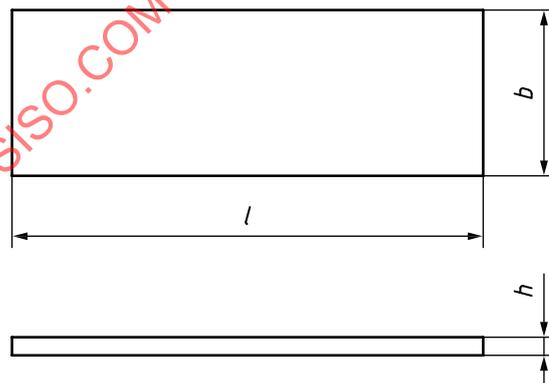
Key

b breadth of (11 ± 1) mm

h thickness (height) of (1,2 ± 0,2) mm

l length of (31 ± 1) mm

Figure 3 — Dimensions of specimens for flexure and acoustic resonance methods

The broad faces and long edges shall be flat and parallel according to the following criteria:

— Flatness: The inner and outer minimum zone references planes (see ISO 12781-1 and Figure 4) of the largest faces shall have a separation Δh less than 0,01 mm. For the long edge faces, the separation of the inner and outer minimum zone reference planes shall be less than 0,1 mm.

— Parallelism: The greatest and least measured thicknesses h_1 and h_2 (see Figure 4) shall agree to within 0,01 mm. For the long-edge faces, the dimensional measurements shall agree to be within 0,1 mm.

7.5.4 Acoustic resonance method

For the acoustic resonance method, prepare three specimens with dimensions of approximately 34 mm × 13 mm × 1,5 mm and grind, finishing with P1200 wet silicon carbide paper complying with ISO 6344-3, to obtain dimensions that comply with Figure 3.
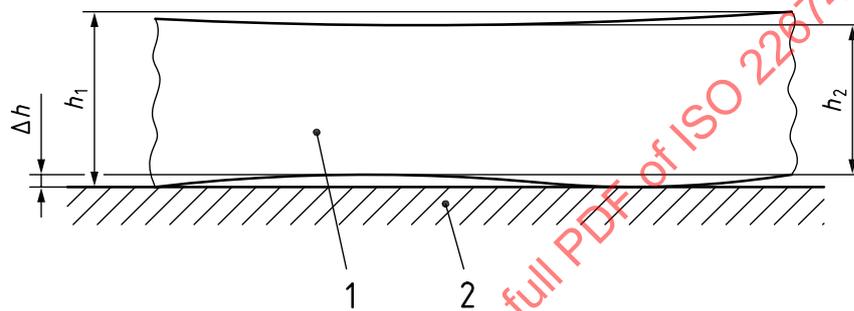
Key

1   specimen
2   flatness reference plane
Δh separation of outer and inner reference planes
h_1 greatest measured thickness
h_2 least measured thickness

Figure 4 — Specimen for acoustic resonance method and reference planes

The faces shall be rectangular, flat and parallel according to the following criteria.

— Rectangular: The angles between faces and edges shall be (90 ± 1)°.

— Flatness: The inner and outer minimum zone reference planes (see ISO 12781-1:2011 and Figure 4) of the largest faces shall have a separation Δh less than 0,01 mm. For edge faces, the separation of the inner and outer minimum zone reference planes shall be less than 0,1 mm.

— Parallelism: The greatest and least measured thicknesses h_1 and h_2 (see Figure 4) shall be within 0,01 mm. For the edge faces, the dimensional measurements shall be within 0,1 mm.

7.6 Density measurement

7.6.1 Solid material

Use material taken from retail packages without further processing. Process the material according to the instructions for use, given in 10.2. Prefer a regular shape and geometry for the specimen if possible, for example, rod. With irregularly shaped material choose pieces on which all parts of the external surfaces are visible.

7.6.2   Powder material

For materials for additive manufacturing, fabricate five (5 ± 0,01) mm diameter cylindrical pieces (5 ± 0,01) mm long.

7.7   Corrosion resistance

Prepare two rectangular samples with dimensions of approximately 34 mm × 13 mm × 1,5 mm, formed, finished and heat-treated in accordance with 7.1 and 7.2. Polish to the finish specified by the manufacturer according to 10.2 a).

If the processing technique or material itself does not allow the production of samples with a rectangular shape and dimensions 34 mm × 13 mm × 1,5 mm, prepare multiple samples which provide approximately the same surface area (10,2 cm^2).

For semi-finished products, prepare two samples that have approximately the same surface area (10,2 cm^2). These samples are not restricted to the rectangular sample geometry.

If the product is intended for use both with and without a ceramic veneer, two sets of two specimens are required, one to be treated in accordance with 7.2.3 and the other to be treated in accordance with 7.2.2 or 7.2.4 (as applicable).

7.8   Tarnish resistance and colour

To test for cyclic tarnish resistance in accordance with 8.8, prepare specimens in accordance with 7.1, 7.2 and ISO 10271:2020, 4.3.6.

To test for static tarnish resistance in accordance with 8.9, prepare specimens in accordance with 7.1, 7.2 and ISO 10271:2020, 4.4.6.

7.9   Linear thermal expansion

Prepare two rod or bar shaped specimens with a cross-sectional shape and length appropriate to the dilatometer to be used and a cross-sectional area no greater than 30 mm^2. Form, finish and heat treat the specimens in accordance with 7.1 and 7.2.

8   Measurement and test methods

8.1   Information, instructions and marking

Visually inspect to check that the requirements specified in Clause 10 have been met.

8.2   Chemical composition

Analyse quantitatively for all alloying elements that are declared in accordance with 10.1 a). Use analytical procedures with an accuracy and sensitivity that produces a value accurate to a mass fraction of 0,1 % for elements that are present in excess of a mass fraction of 1,0 % and for nickel in excess of a mass fraction of 0,1 %, if applicable. Record the method used for each element. Record and report the mass fraction of each element as a percentage.

If any element that is not a declared alloying element [see 10.1 a)] is detected and present in a concentration greater than or equal to a mass fraction of 0,1 %, record and report it.

Analyse quantitatively for beryllium, cadmium, lead and nickel [if nickel is not listed in 10.1 a)]. Use analytical procedures with an accuracy and sensitivity that produces a value accurate to a mass fraction of 0,01 %. Record the method used for each element and its concentration in the alloy as a mass fraction percentage. If the cadmium and beryllium and lead contents are less than the permitted

limit (see 5.2.2) and that for nickel is less than a mass fraction of 0,1 % (see 5.2.3), report each as "not present". If the contents are higher than the permitted limits, record and report it.

If nickel is detected in excess of a mass fraction of 0,1 %, report this value to an accuracy of a mass fraction 0,1 %.

8.3 Mechanical testing

8.3.1 Apparatus

8.3.1.1 Micrometer, accurate to 0,005 mm with a measuring capacity of 25 mm.

8.3.1.2 Mechanical testing machine (with suitable capacity for both frame and load cell), with an accuracy better than 1 % of the measured value and a cross-head speed up to 2 mm/min. Conduct tensile testing with grips appropriate for the specimens to be used.

8.3.1.3 Extensometer, of class 1 according to ISO 9513, capable of measuring to an accuracy of 1 µm and compatible with the specimen gauge length.

8.3.1.4 Travelling microscope, with at least ×10 magnification, accurate to 0,01 mm with a measuring capacity of 25 mm. An alternative instrument for measuring the gauge length can be used in place of the travelling microscope if it has an accuracy of 0,02 mm or better.

8.3.2 Test procedure

Measure the diameter of the test specimen within the gauge length portion to an accuracy of 0,01 mm using the micrometer (8.3.1.1). Measure the gauge length (defined by the fine marks or scribed lines) to an accuracy of 0,02 mm, using the travelling microscope (8.3.1.4) or an extensometer (8.3.1.3).

Load the test specimen in tension in the mechanical testing machine (8.3.1.2) at a cross-head speed of (1,5 ± 0,5) mm/min until the specimen fractures. Use the extensometer (8.3.1.3) to measure the extension of the specimen over the extensometer-defined gauge-length to obtain a continuous record free from the effect of testing machine compliance.

Inspect the fractured specimen using eyesight with nominally normal vision without magnification to determine whether a visible defect, either external or internal, contributed to the fracture and whether the fracture occurred within the marks or scribe lines that define the gauge length on the specimen [corrective (non-magnifying) lenses can be worn]. If a visible defect is detected or if the fracture has taken place outside the gauge length, reject the specimen and its result. Take a replacement specimen from the second lot of specimens and repeat the test.

Put the broken parts back together and re-measure the gauge length to an accuracy of 0,02 mm, using the travelling microscope. Record this value.

Alternatively, determine the extension after fracture as the extension registered by the extensometer or by the cross-head position for a load that is 50 % of the maximum load registered for the specimen.

Test no more than 12 specimens.

8.3.3 Proof stress of 0,2 % non-proportional extension

8.3.3.1 Calculation

For each specimen, from the force-extension data, determine the force at which there is 0,2 % non-proportional extension and calculate the proof stress, $R_{p0,2}$, by dividing this force by the original cross-section area within the gauge length of the test piece. Determine the proof stress of 0,2 % non-proportional extension on six specimens prepared in accordance with 7.3 (first test series). Use

specimens from the first lot of six specimens and, if necessary, test some of the specimens from the second lot (as replacements for rejected first lot specimens). If necessary, test the remaining specimens in the second lot of six specimens (second test series).

Report the 0,2 % proof stress of non-proportional elongation, $R_{p0,2}$, for all specimens that were tested and not rejected following the post-test inspection. Report the results to the nearest 1 MPa.

8.3.3.2   Mean value

Calculate the proof stress for 0,2 % non-proportional extension as the mean of the values of all specimens tested and not rejected following the post-test inspection. Report the value to the nearest 5 MPa.

8.3.4   Percentage elongation after fracture

8.3.4.1   Calculation

Determine the percentage elongation after fracture for the same specimens tested in 8.3.2 which meet the requirement for proof stress for 0,2 % non-proportional extension.

If the extensometer returns the extension for 50 % drop in load after maximum, calculate the elongation after fracture as this extension divided by the initial gauge length, expressed as a percentage. If the calculation is automated in the control software of the mechanical test machine, record the criteria that the algorithm employs to define fracture.

Calculate the elongation after fracture by taking the difference between the gauge length readings before and after testing as percentage of the original gauge length. Record this value to the nearest 0,1 %.

Either use the automatic measurement of extension with the extensometer or calculate the elongation after fracture by taking the difference between the readings of the gauge length or the cross-head position before and after testing as the percentage of the original gauge length. Record this value to the nearest 0,1 %.

Report the individual values of the percentage elongation after fracture for all specimens that were tested and not rejected following the post-test inspection.

8.3.4.2   Mean value

Calculate the percentage elongation after fracture as the mean of the values of all specimens tested and not rejected following the post-test inspection. Report the mean value to the nearest 1 %.

8.4   Elastic modulus measurement

8.4.1   Tensile strain method

8.4.1.1   Apparatus

8.4.1.1.1   Micrometer (as in 8.3.1.1).

8.4.1.1.2   Mechanical testing machine (as in 8.3.1.2).

8.4.1.1.3   Extensometer, of class 1 according to ISO 9513, capable of measuring to an accuracy of 1 µm and with a gauge length of at least 10 mm.

8.4.1.2 Measurement and calculation of elastic modulus for tensile strain method

Measure the cross-sectional area of the specimen at each end and at the middle of the gauge length. Perform sufficient measurements (three at 60°) at each position to detect and correct for irregularity. Record the average for the three positions. Load the specimen to a force level that corresponds to no more than 60 % of the 0,2 % proof stress of non-proportional extension [as calculated from 10.1 c), the stated proof stress] and record the force and elongation. Unload to a force to 5 % of the 0,2 % proof stress of non-proportional extension. Undertake this procedure at least four times.

$$E = \frac{\Delta F}{S} \cdot \frac{l_g}{\Delta L} \tag{1}$$

where

E is the elastic modulus, in GPa;

ΔF is the change in the force, in N;

S is the cross-sectional area, in mm^2;

l_g is the initial gauge length, in mm;

ΔL is the extension measured in mm;

Record the average of the most consistent three measurements.

Repeat the measurements on two further specimens.

Report the elastic modulus and its uncertainty, calculated according to B.2.1 for all specimens that were tested to the nearest 1 GPa.

8.4.1.3 Static determination of elastic modulus

For the determination of the elastic modulus the slope should be determined in a range of strain of 10 % to 60 % of the 0,2 % non-proportional expansion curve, to avoid indeterminacies at the beginning and at the end of the straight segment of the curve.

This procedure is recommended for Type 5 non-precious metallic materials.

Determine the elastic modulus using the extensometer recording for extension and the testing machine recording the force. Report the results to the nearest 1 GPa.

8.4.2 Flexure method in three- or four-point bending mode

8.4.2.1 Apparatus

8.4.2.1.1 Micrometer (as in 8.3.1.1).

8.4.2.1.2 Mechanical testing machine (as in 8.3.1.2).

8.4.2.1.3 Flexure jig, with either roller or semi-cylindrical supports of length equal to or greater than the breadth of specimen (see 7.5.3), diameter 1,5 mm ± 0,5 mm and separated by at least 20 mm and not more than 30 mm. For load application, the piston shall have one roller (for three-point) or two rollers (for four-point, see Figure 5) or semi-cylindrical anvils of diameter 1,5 mm ± 0,5 mm. For four-point bending, the separation L_i of the lines of contact with the specimen shall be one-half the separation L_o of the lines of contact of the supports. All lines of contact shall be parallel to within 0,05 mm as measured at the edges of the widest specimen. The separations L_i and L_o shall be recorded to a precision of 0,1 mm.

8.4.2.1.4   Displacement sensor, capable of measuring displacements to within 1 μm. For four-point bending, the sensor arm shall be in light contact under the specimen. For three-point bending, deflection can alternatively be measured by the load piston displacement relative to the support-frame of the jig.
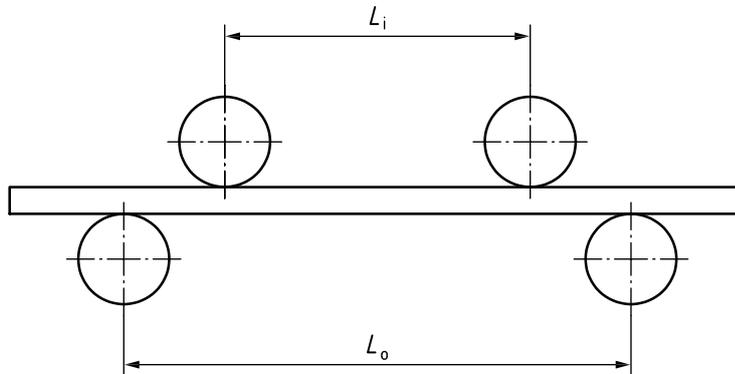
Key

L_i inner separation of load rollers

L_o outer separation of support rollers

Figure 5 — Configuration for four-point bending test

8.4.2.2   Measurement and calculation of elastic modulus by flexure method

Measure and record the breadth, b, of the specimen at five positions along the supported section of the specimen. Measure and record the thickness, h, at 10 well-separated points also within the supported section of the specimen and record the mean and its standard error. Compute the force, F_{60}, required to bring the most highly stressed part of the specimen to 60 % of the material's proof stress of non-proportional extension $R_{P0,2}$ using the appropriate expression in Table 4.

Load the specimen to F_{60}, in the testing machine, recording the deflection, d, as obtained by the jig sensor and force F obtained from load cell. Unload to a force corresponding to 5 % of the 0,2 % proof stress of non-proportional extension F_5. Undertake this procedure at least four times. Compute the apparent elastic modulus E_a for each loading and record the average of the three most consistent values.

NOTE   It is often observed that the initial application of the loading force progressively deforms the surface roughness of the specimen resulting in a lower modulus. On successive loading cycles, values agree within the consistency expected from measurement error estimation.

Calculate the elastic modulus by correcting the value of the apparent elastic modulus obtained with the factor obtained from Table 4 and Table 5 and a suitable estimate of the Poisson's ratio, v. Where data are lacking, assign $v = 0{,}34$ and an uncertainty of 0,04.

Perform this measurement procedure on the three specimens.

To extend the measurement to a determination of the Poisson's ratio, follow the procedure given in Clause C.3.

Table 4 — Expressions for calculating elastic modulus from 3-point and 4-point flexure

Method	3-point flexure	4-point flexure
Maximum load	$F_{60} = \dfrac{0,2R_{p0,2}bh^2}{L_o}$	$F_{60} = \dfrac{0,1R_{p0,2}bh^2}{L_o - L_i}$
Apparent elastic modulus	$E_a = \dfrac{FL_o^3}{4dbh^3}$	$E_a = \dfrac{F(2L_o^3 - 3L_oL_i^2 + L_i^3)}{4dbh^3}$
Correction for anti-clastic bending	$E = E_a \cdot \left[1 - f(b/h)v^2\right]$	$E = E_a \cdot \left[1 - f(b/h)v^2\right]$

The value of the function $f(b/h)$ can be read from Table 5. $R_{p0,2}$ is obtained according to 8.3.3.

Table 5 — Function $f(b/h)$ for calculating the correction for elastic modulus from 3-point and 4-point flexure method

Values and resulting function							
b/h	0,75	0,80	0,85	0,90	0,95	1,0	1,1
$f(b/h)$	0,036 7	0,047 7	0,060 0	0,073 5	0,088 1	0,103 8	0,137 4
b/h	1,2	1,3	1,4	1,5	1,6	1,7	1,8
$f(b/h)$	0,173 3	0,210 6	0,248 4	0,286 2	0,323 5	0,359 7	0,394 8
b/h	1,9	2,0	2,2	2,4	2,6	2,8	3,0
$f(b/h)$	0,428 4	0,460 5	0,519 8	0,572 6	0,619 2	0,660 1	0,695 8
b/h	3,2	3,4	3,6	3,8	4,0	4,4	4,8
$f(b/h)$	0,727 0	0,754 2	0,778 0	0,798 8	0,817 0	0,847 2	0,870 9
b/h	5,2	5,6	6,0	6,5	7,0	7,5	8,0
$f(b/h)$	0,889 6	0,904 6	0,916 8	0,929 0	0,938 8	0,946 7	0,953 1
b/h	9,0	10,0	11,0	12,0	13,0	14,0	15,0
$f(b/h)$	0,963 0	0,970 0	0,975 2	0,979 2	0,982 2	0,984 7	0,986 7
NOTE Linear interpolation between the tabulated values introduces an error no greater than 1 part in 1 000 in the correction factor $[1 - v^2 \cdot f(b/h)]$.							

8.4.2.3   Calculation of the mean value for the elastic modulus

Calculate and report the elastic modulus as the mean of the values of the three specimens tested. Report the standard error in the mean from the nine individual measurements that contribute to the average together with the estimated value as evaluated by the error budget according to Clause B.1.

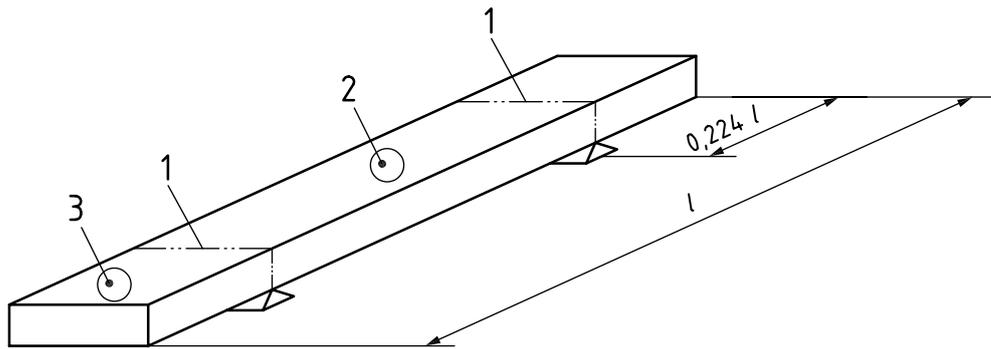### 8.4.3   Acoustic resonance method

8.4.3.1   Apparatus

8.4.3.1.1   Two mounting jigs, for flexure and torsional excitation according to the dimensions shown in Figure 6 and Figure 7.

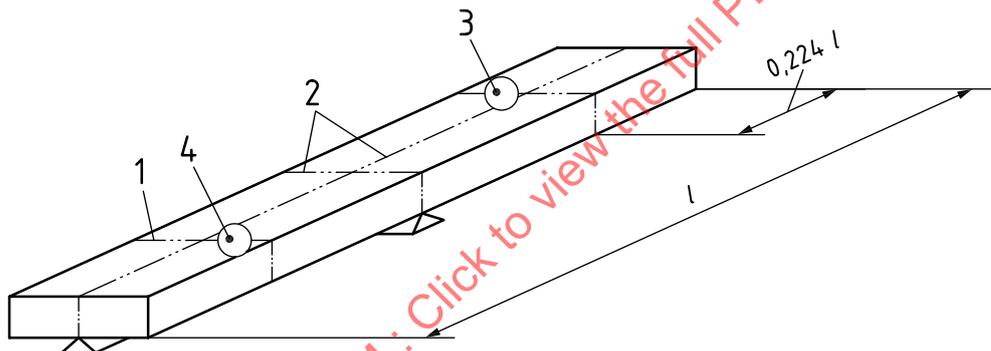**8.4.3.1.2   Micrometer** (as in 8.3.1.1).

8.4.3.1.3 Acoustic audio sensor, with a measurement uncertainty of 5 %.

Key

1   flexure node line
2   impulse point
3   acoustic audio sensor point
l   length (see Figure 3)

Figure 6 — Specimen oriented on the support for flexural vibration

Key

1   flexure node line
2   torsion node line
3   impulse point
4   acoustic audio sensor point
l   length (see Figure 3)

Figure 7 — Specimen oriented on the support for torsional vibration

8.4.3.2   Measurement and calculation of elastic modulus for acoustic resonance method

Measure the dimensions of the specimen to a precision of 0,01 mm. Weigh the specimen to a precision of 0,1 g to find its mass, m. Mount the specimen on a jig and excite it in each of the fundamental flexural and torsional modes of vibration by striking at the points shown in Figure 6 and Figure 7. For each mode, determine the frequency of vibration by means of an acoustic sensor placed as shown in Figure 6 and Figure 7. Repeat the measurement until five readings that agree within 1 % are obtained consecutively. Repeat the measurement on two further specimens.

8.5 Calculation of elasticity parameters from acoustic measurement

8.5.1 General

The calculations require an initial estimate of the Poisson's ratio. The value $v = 0,34$ shall be taken as an initial value and subsequently adjusted according to 8.5.4.

8.5.2 Elastic modulus

Calculate the correction factor C_1 using

$$C_1 = 1 + 6,585(1 + 0,075\,2v + 0,810\,9v^2)\left(\frac{h}{l}\right)^2 - 0,868\left(\frac{h}{l}\right)^4 - \frac{8,34\left((1 + 0,202\,3v + 2,173v^2)\left(\frac{h}{l}\right)^4\right)}{1 + 6,338\left((1 + 0,140\,8v + 1,536v^2)\left(\frac{h}{l}\right)^2\right)}$$

(2)

where

    h is the thickness of the specimen;

    l is the length of the specimen.

NOTE   See Figure 3.

Denote the fundamental frequency measured in the flexural mode of vibration by f_1. Calculate the elastic modulus using

$$E = 0,946\,5\frac{mf_1^2}{b}\left(\frac{l}{h}\right)^3 C_1$$

(3)

where m is the mass of the specimen.

Report the elastic modulus and its uncertainty, calculated according to Clause B.4. Report the value to the nearest 1 GPa.

8.5.3 Shear modulus

Calculate the correction factor C_2 using

$$C_2 = \frac{\left(\frac{b}{h}\right)^2 + 1}{4 - 2,52\left(\frac{h}{b}\right) + 0,21\left(\frac{h}{b}\right)^5}$$

(4)

Denote the fundamental frequency measured for the torsional mode of vibration by f_2. Calculate the elastic shear modulus using

$$G = \frac{4lmf_2^2}{bh}C_2$$

(5)

8.5.4 Poisson's ratio

Calculate the Poisson's ratio using

$$v = \frac{E}{2G} - 1 \tag{6}$$

If the value obtained for the Poisson's ratio differs from the estimate used to compute the correction factors by more than the uncertainty calculated using Formula (B.8), then recalculate the correction factors and iterate the calculations in 8.5.2, 8.5.3 and 8.5.4.

8.6 Density

8.6.1 Preparation of specimen

Determine the density of the metallic material, prepared according to 7.6.

8.6.2 Reagents

8.6.2.1 Water, of grade 3 in accordance with ISO 3696.

8.6.2.2 Ethanol or methanol, analytical grade.

8.6.3 Apparatus

Use a balance, a pycnometer or other suitable device, having an opening and volume appropriate for the specimens to be used.

8.6.4 Procedure

For pieces of regular geometry, the density can be determined by a simple measure and weigh procedure with an appropriate accuracy.

For irregular shaped pieces, immerse the specimens in ethanol or methanol (8.6.2.2) and clean for 2 min in an ultrasonic bath. Rinse the specimens in water (8.6.2.1) and dry with water-free and oil-free compressed air. Clean sufficient material to provide five specimens suitable for the apparatus.

Use the apparatus (e.g. pycnometer, see 8.6.3) and test procedure with an appropriate accuracy, to determine the density of five specimens of metallic material to an accuracy of 0,1 g cm^{-3}.

Report the average value rounded to 0,1 g cm^{-3}.

8.7 Corrosion resistance by the static 7 d immersion procedure of ISO 10271

8.7.1 Preparation of specimen

Use specimens produced in accordance with 7.7.

8.7.2 Reagents

Use reagents according to ISO 10271:2020, 4.1.3.

8.7.3 Apparatus

Use apparatus according to ISO 10271:2020, 4.1.4. The sensitivity of the water-pH meter should be ±0,05 pH units.

8.7.4 Test solution

Prepare a fresh solution for each test according to ISO 10271:2020, 4.1.5.

8.7.5 Test procedure

Determine the surface area of each sample (see 7.7) to the nearest 0,1 cm^2. Immerse the samples in ethanol or methanol (8.6.2.2) and clean for 2 min in an ultrasonic bath. Rinse the samples in water (8.6.2.1) and dry them with water-free and oil-free compressed air. Now, follow the test procedure according to ISO 10271:2020, 4.1.7.

In parallel with the production of sample solutions, produce a reference solution to establish the impurity level for each element in the solution. Take a third glass container and add approximately the same volume of solution as that used to produce the sample solutions. Record the volume used, to an accuracy of 0,1 ml. Close the container to prevent evaporation. Hold at (37 ± 1) °C for 7 d ± 1 h.

8.7.6 Analysis

Use an instrumental analytical method of adequate sensitivity (e.g. AAS or ICP-OES). Analyse the solution quantitatively for those elements that are declared in 10.1 a), for the hazardous elements listed in 5.2.1 and any other metallic element found to be present in a concentration greater than a mass fraction of 1,0 % during the chemical composition analysis of the material (see 8.2).

8.7.7 Treatment of data

For each element, subtract the value obtained for the element in the reference solution from the value obtained in the test solution. For each element, divide the measured metal ion release by the surface area of the specimen or sample (7.7). Report the value for each element in "μg cm^{-2} in 7 d". Use the sum of these values to obtain the total metal ion release. Average the results obtained from the two specimens. Report this average.

If the product is intended for use as both a metallic material without a ceramic veneer and a metal-ceramic material, calculate the metal ion release for each condition and record separately.

8.8 Sulfide tarnish test — Cyclic immersion

Follow the test method given in ISO 10271:2020, 4.3.

8.9 Sulfide tarnish test — Static immersion

Follow the test method given in ISO 10271:2020, 4.4.

8.10 Solidus and liquidus temperatures (dental casting alloys) or melting point (commercially pure metals)

8.10.1 Cooling curve method

8.10.1.1 General

Heat and melt a suitable amount of the metallic material in a crucible with which it will not react. Use a calibrated temperature sensor that is suited to the expected solidification interval and that will not react with the metallic material. Insert this into the melt at the crucible wall. Remove the melt from the source of heat. Record the temperature over time until the temperature has reached room temperature. Identify the feature in the temperature-time cooling curve that corresponds to the first solidification and determine the associated temperature. Assign this temperature as the liquidus temperature.

Repeat the measurement with the temperature sensor inserted into the middle of the melt. Identify the feature that represents the final solidification and determine its temperature; assign this temperature as the solidus temperature.

Iterate these procedures until three temperature determinations are consistent within a range of ±10 °C for metallic materials with solidus or melting point temperatures below or equal to 1 200 °C, or a range of ±25 °C for metallic materials with solidus or melting point temperatures above 1 200 °C.

8.10.1.2 Further investigations

Where identification of the solidus temperature is confused by further (solid phase) reactions, it is advisable to investigate these metallographically. Retain the solidified specimen. Prepare a new melt with a temperature sensor inserted in the middle and remove from the heat. Follow the cooling of the specimen until the middle of the solidification temperature range and then quench the crucible and semi-molten specimen in a suitable coolant. Prepare the two solidified melts for metallographic examination and identify the phases that have precipitated in the original specimen near the temperature sensor. Use this information to either resolve the solidus temperature, or to determine that thermal analysis (see 8.10.2) is required to complete the determination.

8.10.2 Thermal analysis method

8.10.2.1 Apparatus

A differential thermo-analytic instrument that has been calibrated for the expected solidification temperature range, that can measure the heat of reaction directly, for example, differential scanning calorimetry (DSC), or the consequential temperature change, for example, differential thermal analysis (DTA), and that can be heated and cooled at rates that differ by a factor of 5.

A balance that is able to weigh the specimen to a relative precision of 1 %.

8.10.2.2 Measurement

Load the weighted specimen and its crucible into the instrument.

Run the analyser instrument through a heating and cooling cycle to 100 °C above the expected melting point, recording the differential signal. Retain the specimen for metallographic examination.

Weigh a new specimen and repeat the measurement at heating and cooling rates that differ by a factor of at least 5 from the first run. Retain the specimen.

NOTE   By running the measurement at two different heating and cooling rates, for example, 1 °C/min and 5 °C/min or 2 °C/min and 10 °C/min, the effects of melting and solidification can be distinguished from solid-state phase transitions that occur in complex alloys. Furthermore, the retained specimens will exhibit different microstructures that further aid the identification of the solidus temperature.

8.10.2.3 Interpretation

On the records of both runs, identify the features that correspond to melting and solidification. If solid phase reactions confuse the identification of the melting or solidification range, metallographic examination of the solidified specimens shall be undertaken to identify the amount and size of precipitated phases. This information shall be used to separate the thermal contribution of the solid phase reactions from that of the melting or solidification reactions.

Once the solid-liquid transformation has been resolved from any confusing reactions, assign the temperature at which melting begins on heating as the solidus temperature and that at which solidification begins on cooling as the liquidus temperature.

Repeat these procedures until three temperature determinations are consistent within a range of ±10 °C for metallic materials with solidus or melting point temperatures below or equal to 1 200 °C, or a range of ±25 °C for metallic materials with solidus or melting point temperatures above 1 200 °C.

8.11 Linear thermal expansion

Use specimens of the type described in 7.9.

Determine the linear thermal expansion using a calibrated dilatometer.

Heat at a rate of (5 ± 1) °C per min from room temperature up to 550 °C and record expansion continuously as a function of temperature.

Calculate the coefficient of linear thermal expansion α between 25 °C and 500 °C or between 50 °C and 500 °C for each specimen from the linear expansion/temperature recordings.

NOTE   Two values of the lower point of temperature range are permitted to allow for differences in ambient temperatures.

Report each value in 10^{-6} K^{-1} to the nearest $(0,1 \times 10^{-6})$ K^{-1}.

If seen, report deviations from linear behaviour.

9   Test report

At the conclusion of testing, a test report shall be compiled, presenting the test procedures listed in this document. This report shall include at least the following details:

a)   brand or product name of tested material;

b)   name of the manufacturer;

c)   lot number of the tested material;

d)   information how specimen were produced, for example, by casting, additive manufacturing, subtractive manufacturing;

e)   used materials and devices;

f)   conducted heat treatments (e.g. simulated ceramic firings, stress relieving);

g)   single values and descriptive statistics (e.g. mean value, min/max value, standard deviation);

h)   determination of compliance with the requirements;

i)   density: mean value of five specimen in g/cm^3;

j)   composition: give values with an accuracy of a mass fraction of 0,1 % for all given constituents; give values with an accuracy of a mass fraction of 0,01 % for hazardous elements listed in 5.2;

k)   corrosion: value of all released ions in $\mu g/cm^2$ in 7 d;

l)   tarnish resistance;

m)   solidus/liquidus temperatures or melting point in °C;

n)   proof stress for 0,2 % of non-proportional extension: report the mean value to the nearest 5 MPa;

o)   elongation after fracture: report the mean value to the nearest 1 %;

p)   elastic modulus: report the mean value to the nearest 1 GPa;

q)   date of test;

r)   name of test organization;

s)   the International Standard used (including its year of publication);

t)   the method used (if the standard includes several);

u)   the result(s), including a reference to the clause which explains how the results were calculated;

v)   any deviations from the procedure;

w)   any unusual features observed.

10 Information and instructions for use

10.1 Information

The following information shall be included in the literature that shall accompany the package.

a)   Composition of the metallic material.

   1)   Each element that is present as an element in a mass fraction greater than 0,1 % shall be declared.

   2)   Each element that is present as an element in a mass fraction greater than 1,0 % shall also have the mass fraction given as a percentage to an accuracy of 0,1 %;

b)   Description of the metallic material using appropriately the definitions and its classification (see 4.2).

   EXAMPLE 1   "Cobalt-based dental alloy, Type 5."

   EXAMPLE 2   "Titanium metal for dental casting, Type 2."

   EXAMPLE 3   "Noble-metal alloy intended for metal-ceramic dental restorations or as a dental casting alloy, Type 3."

c)   0,2 % proof stress of non-proportional extension.

d)   Percentage elongation after fracture.

e)   Elastic modulus.

f)   Density.

g)   If appropriate, the reported tarnish resistance.

h)   Solidus and liquidus temperatures or melting point.

i)   If the product is intended for metal-ceramic restorations, the coefficient of linear thermal expansion.

j)   If the product is intended for use as a casting alloy, the recommended casting temperature.

k)   If the product is intended for metal-ceramic restorations, the highest recommended firing temperature.

l)   Recommendations for brazing, welding or other joining techniques.

m)   Recommendations on storage conditions.

n)   If the product contains nickel in excess of a mass fraction of 0,1 %, adequately detailed information regarding its potential for adverse reactions and the text: 'This product contains nickel'.

o) If the product contains less than or equal to a mass fraction of 0,1 % nickel and the manufacturer intends to label the product "nickel free" [see 10.4 i)], then the following text shall be included: "This product is "nickel free", which means that it contains less than or equal to a Ni mass fraction of 0,1 %".

p) If the product contains less than or equal to a mass fraction of 0,1 % of a specified element [other than one named in 5.2)] and the manufacturer intends to label the product "free of" [see 10.4 k)], then the following text shall be included: "This product is "X free", which means that it contains less than or equal to a mass fraction 0,1 % of X", where X is the name of the specified element.

10.2 Processing instructions

Detailed instructions for use shall be made available to the purchaser by the distributor and/or manufacturer in the literature accompanying the package. These shall include the following:

a) recommendations and information on forming and polishing;

b) if the metallic material is a metal-ceramic metallic material, instructions shall be given on conditioning and for preparing the surface to obtain a satisfactory bond to at least one compatible (named) ceramic material that complies with ISO 9693;

c) if the instructions for use suggest softening or hardening, heat treatment instructions shall be given;

d) advice on protection against dust inhalation shall be given in any recommendation on grinding.

10.3 Marking and labelling

Ingots or blanks shall be marked clearly to identify the manufacturer or supplier, and the product name or an appropriate abbreviation (code).

For metallic materials supplied as small or irregular particles (which cannot be marked directly) or as powder, this requirement is not applicable. Instead, the information shall be available on the packaging which is in direct contact with the metallic material.

10.4 Labelling of the package

The label or insert of the package shall be marked with at least the following information:

a) manufacturer's or authorized representative's name and address;

b) trade name of the metallic material;

c) composition of the metallic material (each element that is present as an element in a mass fraction greater than 1,0 %);

d) description of the product using appropriately the definitions and the Type classification, in Clause 4. This shall be identical to that reported in 10.1 b);

e) lot number;

f) the minimum net mass, expressed in grams (or kilograms);

g) for milling blanks give the dimensions (e.g. diameter, length, breadth, height) and number of pieces in the package;

h) if the metallic material contains more than a nickel mass fraction of 0,1 %, a caution symbol in accordance with ISO 15223-1:2021, Table 1, Reference 5.4.4 (a triangle within which there is an exclamation mark); if the metallic material is supplied in packages in a container, each of the packages shall carry this information; the minimum net mass marked on the package shall be the mass in a single package;

i) if the product contains less than or equal to a nickel mass fraction of 0,1 %, the manufacturer may label the product: "nickel free; contains less than or equal to a nickel mass fraction of 0,1 %";

j) if the product contains less than or equal to a mass fraction of 0,1 % of a specified element (other than one named in 5.2) the manufacturer may label the product: "X free; contains less than or equal to a mass fraction of 0,1 % X", which means that it contains less than or equal to a mass fraction of 0,1 % X, where X is the name of the specified element.

Annex A
(informative)

Tensile testing of a non-cast Type 0 metallic material that is intended for use in a thickness between 0,1 mm and 0,5 mm

A.1 Preparation of test specimen

A.1.1 Geometry, dimensions and tolerances

For metallic materials or semi-finished products for which the processing techniques do not allow the formation of specimen with the shape and dimensions shown in Figure 1 or Figure 2 the geometry, dimensions and tolerances shall be as shown in Figure A.1.

Dimensions in millimetres
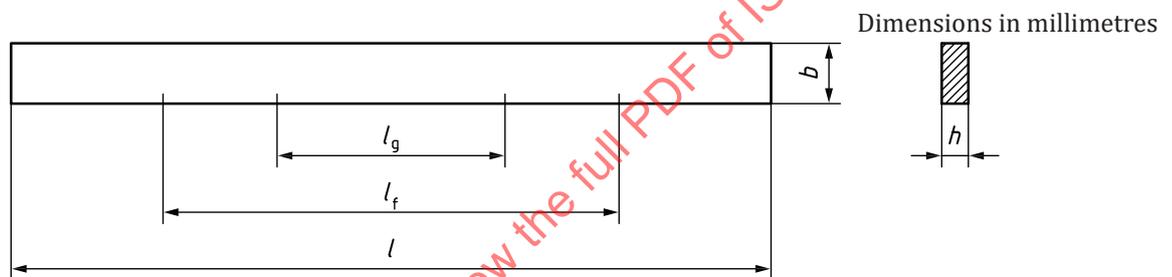
Key

l length, $l > 50$ mm

l_f free length between grips of (30 ± 2) mm

l_g (initial) gauge length of (15 ± 1) mm

b breadth of $(4,0 \pm 0,5)$ mm

h thickness

Figure A.1 — Specimen geometry and dimensions

The thickness h is the thickness recommended by the manufacturer or distributor for the particular application.

The maximum deviation of the breadth b and the thickness h of the specimen along the entire free length between the grips (i.e. within l_f) is 0,05 mm and 0,01 mm, respectively.

A.1.2 Preparation of test pieces by electroforming

Electroform the test specimen with a thickness of 0,2 mm to 0,3 mm, following the instructions for use, as detailed in 10.2. Use dies made in the recommended material (e.g. gypsum, resin, or investment). Remove the die material, separate the connecting material, remove the conducting metal and grind off any overextending metallic material.

Replace any test piece that has a visible defect.

A.1.3 Preparation of test pieces by sintering

Sinter test the material to produce a specimen with a thickness of at least 0,2 mm, following the instructions for use, as detailed in 10.2. Use dies made in the recommended material (e.g. refractory investment). Remove the die material, grind off any overextending metallic material.

Replace any test piece that has a visible defect.

A.1.4 Preparation of test pieces by CAD/CAM techniques

Prepare the test specimen with a thickness of (0,3 ± 0,1) mm, following the instructions for design and fabrication, as detailed in 10.2. Separate the material used for fixing and grind off any overextending metallic material.

Replace any test piece that has a visible defect.

A.2 Number of specimens

Prepare six specimens in accordance with the procedures given in Clause A.1 that will give valid measurements for each material condition in which the metallic material is recommended for use.

NOTE   It can be necessary to prepare one or more additional specimens if any of the results from this set of six specimens are rejected as a consequence of inspection after testing (see A.3.3). The number of additional specimens has to return the number of valid measurements to six.

A.3 Testing

A.3.1 Testing apparatus

A.3.1.1 Micrometer, accurate to 0,005 mm with a measuring capacity of 25 mm.

A.3.1.2 Universal tensile testing machine, of class 1 according to ISO 7500-1.

A.3.1.3 Extensometer, of class 1 according to ISO 9513 with an accuracy better than 1 µm. Consider using a contactless extensometer for any metallic material that has low value for $R_{p0,2}$.

A.3.2 Conditions of testing

Testing shall be done in accordance with ISO 6892-1, if not otherwise specified herein.

a) Hold the specimen in appropriate grips to avoid any bending of the test piece or notching of its surface.

b) Make every endeavour to ensure that test pieces are held in such a way that the force is applied as axially as possible.

A.3.3 Test procedure

For the determination of proof stress of 0,2 % of non-proportional extension ($R_{p0,2}$) and percentage elongation after fracture ($A_{15\,mm}$), produce specimens according to Clause A.1. Test in the as-processed state following any heat treatment (if appropriate) recommended by the manufacturer for a particular application (according to 10.2).

Calculate the original cross-sectional area S_0 within the free length between grips to an accuracy of ±5 %.

Conduct the test at ambient room temperature.