INTERNATIONAL STANDARD

ISO 22674

Second edition
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Dentistry — Metallic materials for fixed and removable restorations and appliances

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

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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.

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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This second edition cancels and replaces the first edition (ISO 22674:2006), which has been technically revised with the following changes:

— Corrosion resistance measurement was referred to the recent standard ISO 10271:2011.

— A second tarnish test was included, referring to provisions in ISO 10271:2011.

— Clarification of the term "free of" was added to the requirements of composition and labelling;

— Lead was added as a hazardous element.

— Measurement of elasticity was revised. Beside the method of calculation of elastic modulus using an extensometer, other alternative methods were added, namely, the flexure method in three- and four-point bending and the acoustic resonance method.

— Informative Annex A was added, dealing with tensile testing of non-cast Type 0 metallic materials intended for use in a thickness between 0,1 and 0,5 mm.

— Normative Annex B was added, giving information on calculation of uncertainty for elastic measurement.

— Informative Annex C was added, giving information for measurement of Poisson ratio.

Introduction

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that, in assessing possible biological hazards, reference has to 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 International Standard 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 continue to be specified in ISO 9693-1.

Requirements for the proof stress and minimum elongation after fracture for Type 0 metallic materials are not included in this International Standard, but it is recommended to adopt the test procedure given in Annex A when measuring these properties. Requirements will be included in a revision of this International Standard when information becomes available to Technical Committee ISO/TC 106/SC 2.

Dentistry — Metallic materials for fixed and removable restorations and appliances

1 Scope

This International Standard classifies metallic materials that are suitable for the fabrication of dental restorations and appliances, including metallic materials recommended for use either with or without a ceramic veneer, or recommended for both uses, and specifies their requirements. Furthermore, it specifies requirements with respect to packaging and marking the products and to the instructions to be supplied for the use of these materials, including products delivered for sale to a third party.

This International Standard does not apply to alloys for dental amalgam (ISO 24234), dental brazing materials (ISO 9333), or metallic materials for orthodontic appliances (ISO 15841) (e.g. wires, brackets, bands and screws).

2 Normative references

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

ISO 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[1], *Implants for surgery — Metallic materials — Part 3: Wrought titanium 6-aluminium 4-vanadium alloy*

ISO 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 6892-1[2], *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

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

ISO 9693-1, *Dentistry — Compatibility testing — Part 1: Metal-ceramic systems*

ISO 10271:2011, *Dentistry — Corrosion test methods for metallic materials*

ISO 15223-1:2012, *Medical devices — Symbols to be used with medical device labels, labelling and information to be supplied — Part 1: General requirements*

3 Terms and definitions

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

3.1
base metal
any metallic element with the exception of noble metals (i. e. gold and metals of the platinum group) and silver

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1) To be published.

2) To be published.

3.2
noble metal
gold and metals of the platinum group

[SOURCE: ISO 1942:2009, 2.187]

3.3
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.

3.4
metallic material
material having the properties that are associated with an alloy, noble metal or base metal

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

3.5
casting alloy
metallic material designed for casting into an investment mould

3.6
ceramic veneer
thin ceramic surface layer present on a metallic material restoration to provide an aesthetic effect

3.7
metal-ceramic restoration
dental restoration in which a ceramic veneer is bonded to a metallic material 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.

3.8
metallic base
metal with highest concentration by mass fraction in the alloy

Note 1 to entry: The name of this element shall precede the words "-based metallic material for dental restoration" or "-based dental casting alloy" or "-based dental metal-ceramic material", as is appropriate.

3.9
base-metal alloy
alloy having a base metal as the principal element

3.10
as-cast condition
metallurgical condition of the metallic material 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).

3.11
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

3.12
hardening
heat-treatment of a metallic material producing a condition which provides a higher 0,2 % proof stress than the "as-cast" state

Note 1 to entry: If recommended by the manufacturer, explicit instructions shall be given in the instructions for use.

3.13
softening
heat-treatment of a metallic material producing a condition which provides a lower 0,2 % proof stress than the "as-cast" state

Note 1 to entry: If recommended by the manufacturer, explicit instructions shall be given in the instructions for use.

3.14
one-surface inlay
an 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

3.15
elastic modulus
E
the ratio of elastic stress to elastic strain, also termed tensile elastic modulus or Young's modulus, symbol E

4   Classification

For the purposes of this International Standard, 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, e.g. small veneered one-surface inlays, veneered crowns;

   NOTE   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, e.g. veneered or unveneered one-surface inlays, veneered crowns;

— **Type 2:** for single tooth fixed prostheses, e.g. 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, e.g. 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, e.g. thin removable partial dentures, parts with thin cross-sections, clasps.

NOTE 1   The higher application type can include lower application types.

NOTE 2   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 1,0 % (mass fraction), each constituent element shall be declared by the manufacturer and shall be reported [see 9.1 a)] to a precision of 0,1 % (mass fraction).

Any element that is present in excess of 0,1 % (mass fraction), but not of 1,0 % (mass fraction), shall be identified in [see 9.1 a)] either by name or symbol.

EXAMPLE   If a constituent element is present with 0,6 % (mass fraction), it shall be identified [9.1 a)] either by name or symbol. If a constituent element is present with 22,06 % (mass fraction), it shall be reported [9.1 a)] as 22,1 % [precision of 0,1 % (mass fraction)].

If the metallic material contains less than 0,1 % (mass fraction) of a specified element (other than one named in 5.2), it may be named as "free of" this specified element [see 9.1 p) and 10.2 h)].

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 9.1 a)] is given in Table 1.

Table 1 — Permitted deviation from the reported composition for elements

Alloy	Elemental content	
	1,0 % < mass fraction ≤ 20 %	mass fraction > 20 %
Base-metal alloy	max. 1,0 %	max. 2,0 %
Silver-based and noble metal alloy	max. 0,5 %	max. 0,5 %

5.2 Hazardous elements

5.2.1 Hazardous elements

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

5.2.2 Limits for the hazardous elements cadmium and beryllium

The metallic material shall not contain more than 0,02 % (mass fraction) cadmium or beryllium.

Cadmium and beryllium are neither alloying elements nor elements inherent to the manufacturing process of titanium metallic materials. For titanium metallic materials that comply with the requirements of chemical composition of ISO 5832-2 and ISO 5832-3, cadmium and beryllium do not need to be analysed.

5.2.3 Limit for lead

The metallic material shall not contain more than 0,02 % (mass fraction) lead.

Lead is neither an alloying elements nor an element inherent to the manufacturing process of titanium metallic materials. For titanium metallic materials that comply with the requirements of chemical composition of ISO 5832-2 and ISO 5832-3, lead does not need to be analysed.

5.2.4 Nickel

5.2.4.1 Manufacturer's reported nickel content and permitted deviation

If the metallic material contains more than 0,1 % (mass fraction) nickel, this content shall be given to an accuracy of 0,1 % (mass fraction) in the literature which accompanies the package [see 9.1 n)] and on the package, label or insert [see 10.2 f)].

The mass fraction shall not exceed the value stated in 9.1 n) and 10.2 f).

5.2.4.2 Nickel-free products

For the purposes of this International Standard, alloys with a maximum of 0,1 % (mass fraction) nickel can be labelled "nickel free" [see 9.1 o) and 10.2 g)].

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

5.3 Biocompatibility

For guidance on biocompatibility, see Introduction.

5.4 Mechanical properties

5.4.1 General

This requirement (see Table 2) 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.

If a heat-treatment is recommended by the manufacturer [see 9.2 c)] this requirement shall be met by the material in the heat-treated condition, applied in accordance with the manufacturer's instructions for use.

For a metallic material, the Type can be classified differently (according to Table 2) if it is recommended for use in more than one metallurgical condition (produced by alternative processing schedules). The highest applicable Type shall be specified for each condition.

Table 2 — Mechanical properties

Type	Proof stress of 0,2 % non-proportional extension $R_{p0,2}$ MPa minimum	Elongation after fracture % minimum	Elastic modulus GPa minimum
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 9.1 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 (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 (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 meet the requirement for proof stress of 0,2 % 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 % 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 % 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 (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 % 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 % 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 8.4.2 shall not differ by more than 10 % from the value given in the literature accompanying the package [see 9.1 c)].

5.4.3   Elongation after fracture

5.4.3.1   Determination of compliance with the requirement

Refer to the classification Type stated in 9.1 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 % 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 (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 % non-proportional extension also meet the requirement for elongation after fracture, the metallic material complies with the requirement.

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 % 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 % 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 % 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 must 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 8.5.2 must exceed 70 % of the value stated in the literature accompanying the package and not be less than the minimum value for the type [see 9.1 d)].

5.5   Elastic modulus

5.5.1   Precision of measurement method

The measurement of elastic modulus shall be performed by a method (see 8.6) capable of a precision that gives a combined standard uncertainty less than 10 % of the measured value.

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

5.5.2   Determination of compliance with the requirements for Type 5 materials

Refer to classification Type stated in 9.1 b).

Where compliance with Type 5 is claimed, the mean value of the elastic modulus as determined according to 8.6 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 9.1 e)] by more than 10 %.

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 9.1 f)].

5.7 Corrosion resistance for material integrity

When tested in accordance with 8.9, the total metal ion release from the metallic material into the specified solution (see 8.9.4) 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 8.10 or 8.11, 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 9.1 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 9.1 h)] when tested in accordance with 8.12.

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 9.1 h)] when tested in accordance with 8.12.

For a commercially pure metal, the melting point shall not differ from the value stated on the literature accompanying the package [see 9.1 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 8.12.

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 8.13, 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 9.1 i)].

5.11 Information, instructions and marking

All the requirements specified in Clause 9 and 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 test specimens

7.1 General

Prepare all test specimens following the manufacturer's instructions for use, as detailed in 9.2.

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

Replace any test specimens 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 manufacturer's instructions for use

If a heat-treatment is recommended by the manufacturer [see 9.2 c)], perform the tests in the heat-treated condition in accordance with the manufacturer's 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 9.1 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 manufacturer's 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

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).

Test specimens 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 9.2 a)], record this.

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

Dimensions in millimetres
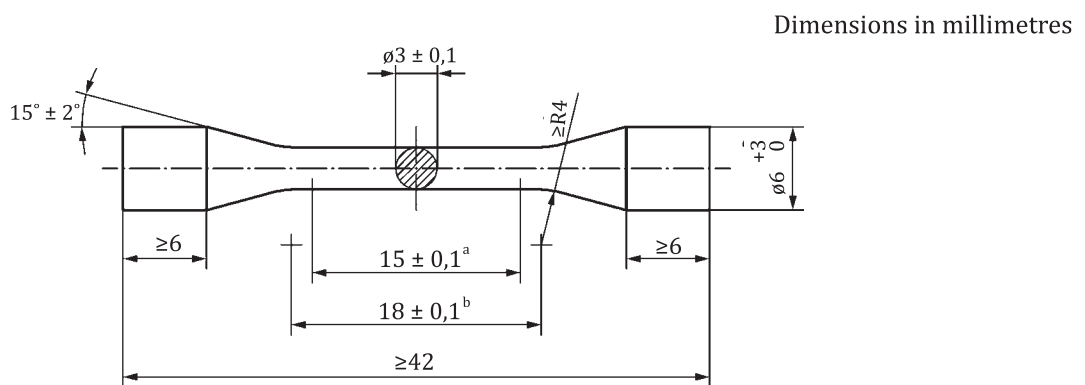
Key

a   gauge length

b   rotation symmentrical section of test specimen

Figure 1 — Test specimen with conical shoulders

Dimensions in millimetres
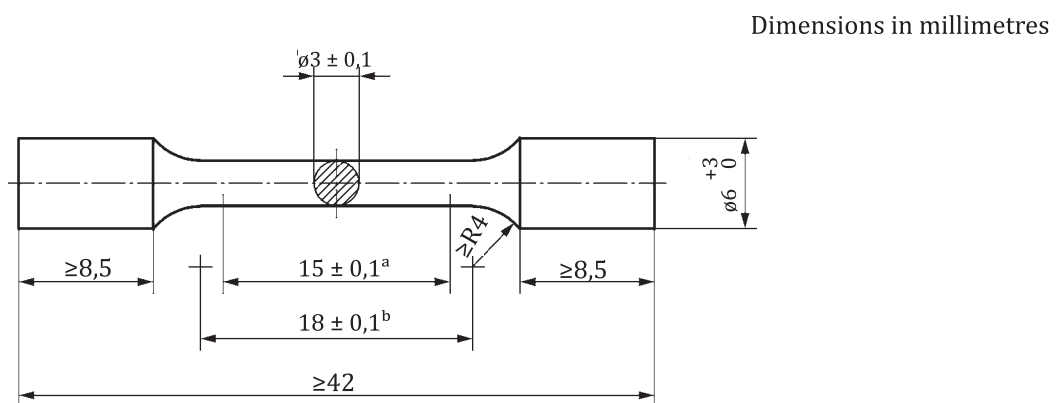
Key

a   gauge length

b   parallel section of test specimen

Figure 2 — Test 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 test specimens 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.

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, A, of the gauge length can be determined to a precision for which the standard uncertainty in A is less than 2 % of the measured value, these specimens can be used directly.

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

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 6344-1 before treatment corresponding to the manufacturer's 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 plate specimens with dimensions of approximately 34 mm × 13 mm × 1,5 mm and grind, finishing with P1200 wet silicon carbide paper complying with ISO 6344-1, to obtain dimensions that comply with Figure 3.

Dimensions in millimetres
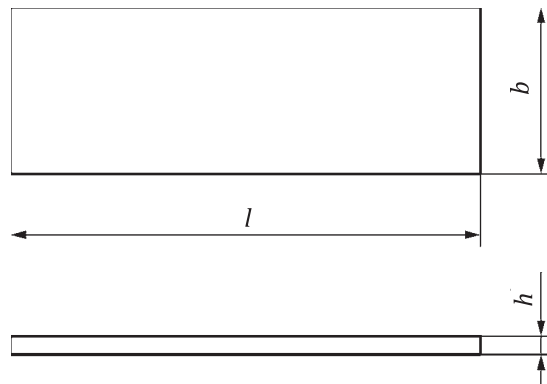
Key

b width = (11 ± 1)

h thickness = $(1,2 \pm 0,2)$

l length = (31 ± 1) mm

Figure 3 — Dimensions of plate specimens for the 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 12781-1:2011 and Figure 4) of the largest faces shall have a separation δh_{max} 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 within 0,1 mm.

7.5.4   Acoustic resonance method

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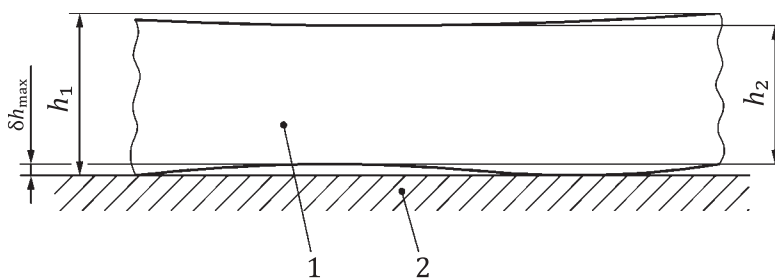
Key

1   specimen plate

2   flatness reference plate

δh_{max} separation of outer and inner reference planes

h_1 greatest measured thickness

h_2 least measured thickness

Figure 4 — Plate specimen for acoustic resonance method, 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 references planes (see ISO 12781-1:2011 and Figure 4) of the largest faces shall have a separation δh_{max} less than 0,01 mm. For edge faces, the separation of the inner and outer MZ 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 edge faces, the dimensional measurements shall agree to within 0,1 mm.

7.6 Density measurement

Use material taken from retail packages without further processing, if it is free from pores. Otherwise, process the material according to the manufacturer's instructions for use, given in 9.2. Prefer a regular shape and geometry for the test specimens if possible, e.g. rod. With irregularly shaped material choose pieces on which all parts of the external surfaces are visible.

7.7 Corrosion resistance

Prepare two rectangular plate specimens 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 9.2 a).

If the processing technique or material itself does not allow the production of test specimens with a rectangular shape and dimensions 34 mm × 13 mm × 1,5 mm, prepare two specimen 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 plate 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 8.10, prepare specimens in accordance with 7.1, 7.2 and ISO 10271:2011, 4.3.6

To test for static tarnish resistance in accordance with 8.11, prepare specimens in accordance with 7.1, 7.2 and ISO 10271:2011, 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 Test methods

8.1 Information, instructions and marking

Visually inspect to check that the requirements specified in Clause 9 and Clause 10 have been met.

8.2 Chemical composition

Analyse quantitatively for all alloying elements that are declared in accordance with 9.1 a). Use analytical procedures with an accuracy and sensitivity that will produce a value accurate to 0,1 % (mass fraction) for elements that are present in excess of 1,0 % (mass fraction) and for nickel in excess of 0,1 % (mass fraction), 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 9.1 a)] is detected and present in a concentration greater than or equal to 0,1 % (mass fraction), record and report this.

Analyse quantitatively for beryllium, cadmium, lead and nickel [if nickel is not listed in 9.1 a)]. Use analytical procedures with an accuracy and sensitivity that will produce a value accurate to 0,01 % (mass fraction). Record the method used for each element and its concentration in the alloy as a % (mass fraction). If the cadmium and beryllium and lead contents are less than the permitted limit (5.2.2) and that for nickel is less than 0,1 % (mass fraction) (5.2.3), report each as "not present". If the contents are higher than the permitted limits, record and report this.

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

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⁻¹. 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 10x 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). Using the travelling microscope (8.3.1.4), measure the gauge length (defined by the fine marks or scribed lines) to an accuracy of 0,02 mm.

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 remeasure the gauge length to an accuracy of 0,02 mm, using the travelling microscope. Record this value.

Test no more than 12 specimens.

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

8.4.1 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 test 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.

8.4.2 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.

8.5 Percentage elongation after fracture

8.5.1 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.

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 %.

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

8.5.2 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 %.

8.6 Elastic modulus measurement

8.6.1 Tensile strain method

8.6.1.1 Apparatus

8.6.1.1.1 Micrometer (as in 8.3.1.1).

8.6.1.1.2 Mechanical testing machine (as in 8.3.1.2).

8.6.1.1.3 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.

8.6.1.2 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 9.1 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.

NOTE   It is frequently 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 with the consistency expected from measurement error estimation.

$$E = \frac{\Delta P}{A} \cdot \frac{L}{\Delta L} \qquad\qquad (1)$$

where

E is the elastic modulus, in GPa;

ΔP is the change in the force, in N;

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

L 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.

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

8.6.2.1 Apparatus

8.6.2.1.1 Micrometer (as in 8.3.1.1).

8.6.2.1.2 Mechanical testing machine (as in 8.3.1.2).

8.6.2.1.3 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 ± 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 ± 0,5 mm. For four-point bending, the separation L_0 of the lines of contact with the specimen shall be one-half the separation L 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 and L_0 are to be recorded to a precision of 0,1 mm.

8.6.2.1.4 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.
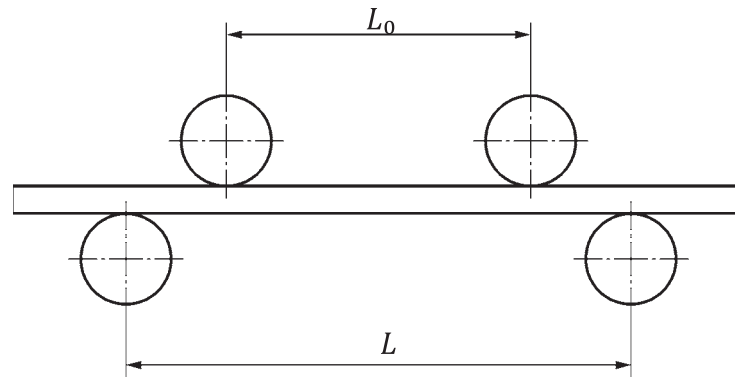
Key

L_0 separation of load rollers

L separation of support rollers

Figure 5 — Configuration for four point bending

8.6.2.2 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, P_{60}, required to bring the most highly stressed part of the specimen to 60 % of the material's proof stress of non-proportional extension $R_{\mathrm{P0,2}}$ using the appropriate expression in Table 3.

Load the specimen to P_{60}, in the testing machine, recording the deflection, d, as obtained by the jig sensor and force P obtained from load cell. Unload to a force corresponding to 5 % of the 0,2 % proof stress of non-proportional extension, P_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 frequently 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 3 and Table 4 and a suitable estimate of the Poisson ratio, ν. Where data is lacking, assign ν = 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 ratio, follow the procedure given in C.3.

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

Method	3-point flexure	4-point flexure
Maximum load	$P_{60} = \dfrac{0,2 R_p bh^2}{L}$	$P_{60} = \dfrac{0,1 R_p bh^2}{L - L_0}$
Apparent elastic modulus	$E_a = \dfrac{PL^3}{4dbh^3}$	$E_a = \dfrac{P(2L^3 - 3LL_0^2 + L_0^3)}{4dbh^3}$
Correction for elastic modulus	$E = E_a \cdot \left[1 - f(b\,/\,h)\nu^2 \right]$	$E = E_a \cdot \left[1 - f(b\,/\,h)\nu^2 \right]$

The value of the function $f(b/h)$ can be read from Table 4. $R_{p0,2}$ is obtained according to 8.4.

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

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 $[1 - v^2 \cdot f(b/h)]$.

8.6.2.3   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 B.1.

8.6.3   Acoustic resonance method

8.6.3.1   Apparatus

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

8.6.3.1.2   Micrometer, (as in 8.3.1.1).

8.6.3.1.3   Acoustic sensor.

Key
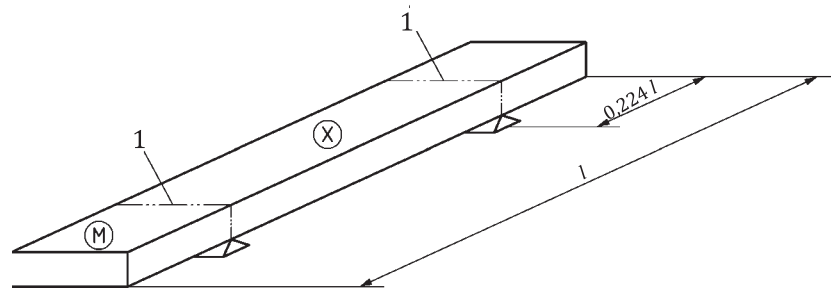
1   flexure node line

X   impulse point

M   microphone sensor point

l   length of the specimen (see Figure 3)

Figure 6 — Specimen oriented on the support for flexural vibration

Key
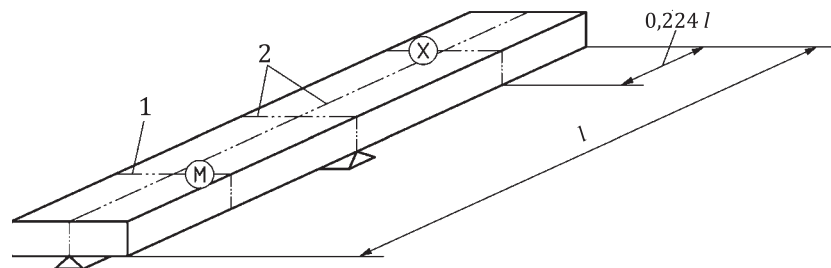
1   flexure node line

2   torsion node line

X   impulse point

M   microphone sensor point

l   length of the specimen (see Figure 3)

Figure 7 — Specimen oriented on the support for torsional vibration

8.6.3.2   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 audio 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.

8.7   Calculation of elasticity parameters from acoustic measurement

8.7.1   General

The calculations require an initial estimate of the Poisson ratio. In want of other information, the value ν = 0,34 shall be taken as an initial value and subsequently adjusted according to C.3.

8.7.2 Elastic modulus

Calculate the correction factor C_1 using

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

(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 B.4. Report the value to the nearest 5 GPa.

8.7.3 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

$$G = \frac{4Lmf_2}{bh}C_2$$

(5)

8.7.4 Poisson ratio

Calculate the Poisson ratio using

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

If the value obtained for the Poisson ratio differs from the estimate used to compute the correction factors by more than the uncertainty calculated using Formula B.9, then recalculate the correction factors and iterate the calculations in 8.7.2, 8.7.3 and 8.7.4.

8.8 Density

8.8.1 Preparation of test specimens

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

8.8.2 Reagents

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

8.8.2.2 Ethanol or methanol, analytical grade.

8.8.3 Apparatus

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

8.8.4 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 (8.8.2.2) and clean for 2 min in an ultrasonic bath. Rinse the specimens in water (8.8.2.1) and dry with water-free and oil-free compressed air. Clean sufficient material to give five samples suitable for the apparatus. Use apparatus (e.g. pycnometer, 8.8.3) and test procedure with an appropriate accuracy, to determine the density of five samples of metallic material to an accuracy of 0,1 g cm^{-3}.

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

8.9 Corrosion resistance by the static immersion procedure of ISO 10271:2011

8.9.1 Preparation of test specimens

Use specimens produced in accordance with 7.7.

8.9.2 Reagents

Use reagents according to ISO 10271:2011, 4.1.3

8.9.3 Apparatus

Use apparatus according to ISO 10271:2011, 4.1.4. The sensitivity of the water-pH meter should be ±0,05 pH units.

8.9.4   Test solution

Prepare a fresh solution for each test according to ISO 10271:2011, 4.1.5.

8.9.5   Test procedure

Determine the surface area of each test specimen or sample ([7.7](#)) to the nearest 0,1 cm^2. Immerse the specimens in ethanol or methanol ([8.8.2.2](#)) and clean for 2 min in an ultrasonic bath. Rinse the specimens in water ([8.8.2.1](#)) and dry them with water-free and oil-free compressed air. Now, follow the test procedure according to ISO 10271:2011, 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.

8.9.6   Analysis

By using an instrumental analytical method of adequate sensitivity (for example, AAS or ICP-OES), analyse the solution quantitatively for those elements that are declared in [9.1](#) a), for the hazardous elements listed in [5.2.1](#) and any other metallic element found to be present in a concentration greater than 1,0 % (mass fraction) during chemical composition analysis of the material ([8.2](#)).

8.9.7   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.

8.10 Sulfide tarnish test (cyclic immersion)

See ISO 10271:2011, 4.3.

8.11 Sulfide tarnish test (static immersion)

See ISO 10271:2011, 4.4.

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

8.12.1 Cooling curve method

8.12.1.1 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.

8.12.1.2 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 (8.12.2) is required to complete the determination.

8.12.2 Thermal analysis method

8.12.2.1 Apparatus

A differential thermo-analytic instrument that has been calibrated for the expected solidification temperature range, that can measure the heat of reaction directly (DSC) or the consequential temperature change (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 %.

8.12.2.2 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.

8.12.2.3 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.

8.13 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 Information and instructions for use

9.1 Information

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

a) the composition of the dental 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) the description of the dental metallic material using appropriately the definitions 3.4, 3.6, 3.7, 3.8 and its classification according to Clause 4. The following Note gives examples of correct description:

   NOTE   "Cobalt-based dental alloy, Type 5."

   "Titanium metal for dental casting, Type 2."

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

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

d) the percentage elongation after fracture;

e) elastic modulus;

f) the density;

g) if appropriate, the reported tarnish resistance (see 5.8);

h) the 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 0,1 % (mass fraction), 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 0,1 % nickel (mass fraction) and the manufacturer intends to label the product "nickel free" [see 10.2 g)], then the following text must be included: This product is "nickel free" which means that it contains less than or equal to 0,1 % (mass fraction) of Ni according to its definition in ISO 22674;

p) if the product contains less than or equal to 0,1 % (mass fraction) of a specified element (other than one named in 5.2) and the manufacturer intends to label the product "free of" [see 10.2 h)], then the following text shall be included: This product is "X free" which means that it contains less than or equal to 0,1 % (mass fraction) of X according to the regulations of ISO 22674, where X is the name of the specified element.

9.2   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 9693-1;

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.

e) marking and labelling

10   Marking and labelling

10.1 Marking

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.

10.2 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 or trademark and address;

b) trade name of the metallic material;

c) description of the product using appropriately the definitions 3.4, 3.6, 3.7, 3.8 and the Type classification, in Clause 4. This is to be identical to that reported in 9.1 b);

d) lot number;

e) the minimum net mass, expressed in grams (or kilograms), or if not applicable (e.g. for milling blanks), give the dimensions (e.g. diameter, length, width, height) and number of pieces in the package;

25

f)   if the metallic material contains more than 0,1 % (mass fraction) nickel, a caution symbol in accordance with ISO 15223-1:2012, Table 1. (5.4.3, a triangle within which there is an exclamation mark) if the metallic material is supplied in packages in a container, each of the packages must carry this information. The minimum net mass marked on the package shall be the mass in a single package;

g)   if the product contains less than or equal to 0,1 % nickel (mass fraction) the manufacturer may label the product "nickel free; contains less than 0,1 % nickel";

h)   if the product contains less than or equal to 0,1 % (mass fraction) 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 0,1 % which means that it contains less than or equal to 0,1 % (mass fraction) X, where X is the name of the specified element.
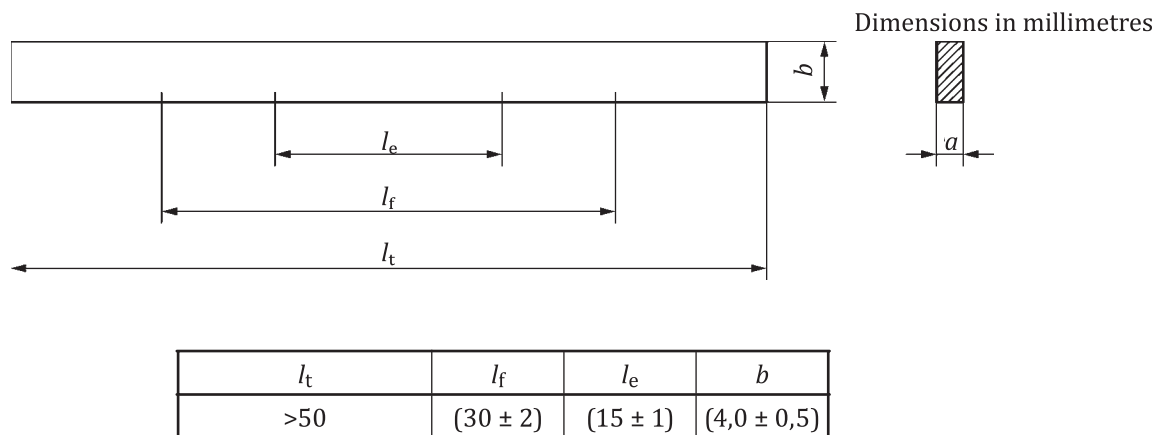
Annex A
(informative)

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

Geometry, dimensions and tolerances shall be as shown in Figure A.1.

Dimensions in millimetres

l_t	l_f	l_e	b
>50	(30 ± 2)	(15 ± 1)	(4,0 ± 0,5)

Key

l_t total length

l_f free length between grips

l_e extensometer gauge length

b specimen width

a specimen thickness

Figure A.1 — Specimen geometry and dimensions

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

The maximum deviation of the width b and the thickness a 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 manufacturer's instructions for use, as detailed in 9.2. 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 manufacturer's instructions for use, as detailed in 9.2. 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 manufacturer's instructions for design and fabrication, as detailed in 9.2. Separate the material used for fixing and grind off any overextending metallic material.

Replace any test piece that has a visible defect.

A.2   Specimen numbers

According to the procedures given in A.1, prepare six specimens that will give valid measurements for each material condition in which the metallic material is recommended for use.

NOTE   It may 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 is 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 $R_{\mathrm{p0,2}}$.

A.3.2   Conditions of testing

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

a)   hold the test specimens 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 % non-proportional extension ($R_{\mathrm{p0,2}}$) and percentage elongation after fracture ($A_{15\ \mathrm{mm}}$), produce specimens according to A.1. Test in the as-processed state following any heat treatment (if appropriate) recommended by the manufacturer for a particular application (according to 9.2).

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.

Use the extensometer to measure the extension of the test specimen over the extensometer gauge length to obtain a continuous record.

Load the specimens in tension in the testing machine, operating at a cross-head speed of $(1,5 \pm 0,5)$ mm/min up to the fracture point of the specimens.

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 gauge length on the specimen. [Corrective (non-magnifying) lenses may 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 and repeat the test.

A.3.4   Determination of proof stress ($R_{p0,2}$) and elongation after fracture ($A_{15\,mm}$)

For each specimen, from its force-extension diagram, determine the force at which there is 0,2 % non-proportional extension and calculate $R_{p0,2}$ by dividing this force by the original cross-sectional area S_0 (see A.3.4) of the test piece. [This property can be obtained, also, without plotting the force-extension curve by using automatic devices (e.g. microprocessor)].

For each specimen, calculate $A_{15\,mm}$ by dividing the permanent elongation of the gauge length by the original extensometer gauge length (l_e, see A.1.1). [This property can be obtained also, without using the continuous record of the extensometer measurement by using automatic devices (e.g. microprocessor). Alternatively, if l_e is defined by fine pencil marks on the surface, direct measurement of the increase in length can be used for the calculation of A_{15mm}].

A.3.5   Evaluation

For each of the six specimen tested (see A.2 and A.3) in each condition (see 9.2), calculate the proof stress of 0,2 % non-proportional extension and the percentage elongation after fracture. Calculate the mean values of these and round them to the nearest 5 MPa or 0,5 %, respectively.

A.4   Test report

The test report shall contain at least the following information:

a)   reference to this International Standard, i.e. ISO 22674;

b)   identification of the test material;

c)   description of the specimen preparation;

d)   description of the test conditions;

e)   measured proof stress of 0,2 % non-proportional extension and percentage elongation after fracture for each specimen and mean values.

Annex B
(normative)

Calculation of uncertainty for elasticity measurement

B.1 Representation of uncertainty for elasticity measurement

Estimation of a test method's capability to attain sufficient precision requires estimates of the uncertainties in the individual quantities that enter into the formulae for evaluating the test quantity. In the following, it is assumed that measured values are distributed randomly and symmetrically about a mean value. When calibration reveals a bias or an asymmetric distribution of readings from a measurement instrument, the source of bias or asymmetry needs to be investigated and corrected before calculating measurement uncertainty. For further guidance, see ISO/IEC Guide 98-3.

Calculate the Type B standard uncertainty in q as

$$u(q) = \sqrt{D^2 + \frac{\sum_{i=1}^{n}(q_i - q)^2}{n(n-1)}} \tag{B.1}$$

where

$u(q)$ is the standard uncertainty in the measurement of q;

D is the minimum difference between two readings that the measurement instrument can discriminate;

n is the number of individual readings;

q is the mean of n individual readings;

q_i is the value of the ith series of readings.

NOTE   For a calibrated measurement instrument, D will be the calibrated precision; otherwise, for an instrument with a scale or digital display, it will be one-half of the smallest scale division.

Denote by $u(q)/q$ the relative standard uncertainty in the quantity q, and quote it as a percentage.

NOTE   For all methods of determining elastic stiffness parameters, specimen dimensions need to be determined with the greatest precision attainable in order to achieve acceptable relative uncertainty.

B.2 Tensile strain method

B.2.1 Elastic modulus

For a rectangular specimen (see Figure 3), calculate the relative uncertainty in cross-sectional area using

$$\frac{u(A)}{A} = \sqrt{\left(\frac{u(b)}{b}\right)^2 + \left(\frac{u(w)}{w}\right)^2} \tag{B.2}$$

where

$u(A)$ is the standard uncertainty in the measurement of cross-section;

    A is the cross-section as measured according to 8.6.1.2;

    $u(b)$ is the standard uncertainty in the measurement of breadth of the specimen;

    b is the breadth of the specimen;

    $u(w)$ is the standard uncertainty in the measurement of width of the specimen;

    w is the width of the specimen.

For a circular specimen, calculate the relative uncertainty in cross-sectional area using

$$\frac{u(A)}{A} = 2\frac{u(r)}{r} \tag{B.3}$$

where

    $u(A)$ is the standard uncertainty in the measurement of cross-section;

    A is the cross-section as measured according to 8.6.1.2;

    $u(r)$ is the standard uncertainty in the measurement of the radius of the specimen;

    r is the radius of the specimen.

Calculate the standard uncertainty in elastic modulus using

$$\frac{u(E)}{E} = \sqrt{2\left(\frac{u(P)}{\Delta P}\right)^2 + \left(\frac{u(A)}{A}\right)^2 + \left(\frac{u(\Delta L)}{\Delta L}\right)^2} \tag{B.4}$$

where

    $u(E)$ is the standard uncertainty in the measurement of elastic modulus;

    E is the elastic modulus;

    $u(P)$ is the standard uncertainty in the measurement of load force;

    ΔP is the change in load force corresponding to the change in gauge length;

    $u(A)$ is the standard uncertainty in the measurement of cross-section;

    A is the cross-section as measured according to 8.6.1.2;

    $u(\Delta L)$ is the standard uncertainty in the measurements of ΔL;

    ΔL is the change in gauge length measured by the extensometer.

B.3   Flexure method

B.3.1   Elastic modulus

Calculate the uncertainty in the separation of the supports $u(L)$ and, for 4-point bending, the standard uncertainty in the separation of the loading anvils, $u(L_0)$ according to B.1.

Calculate the standard uncertainties $u(b)$, $u(h)$ and $u(d)$ according to B.1 and $u(P)/\Delta P$ according to B.2.1.

For 3-point flexure, calculate the relative uncertainty in elastic modulus using

$$\frac{u(E)}{E} = \sqrt{2\left(\frac{u(P)}{\Delta P}\right)^2 + 2\left(\frac{u(d)}{\Delta d}\right)^2 + \left(\frac{u(b)}{b}\right)^2 + 3\left(\frac{u(h)}{h}\right)^2 + 3\left(\frac{u(L)}{L}\right)^2}$$ (B.5)

where

$u(E)$ is the standard uncertainty in the measurement of elastic modulus;

E is the elastic modulus;

Δd is the change in displacement of the mid-point of the specimen corresponding to the change ΔP in the load force.

For 4-point flexure, calculate the uncertainty in elastic modulus using

$$\frac{u(E)}{E} = \sqrt{2\left(\frac{u(P)}{\Delta P}\right)^2 + 2\left(\frac{u(d)}{\Delta d}\right)^2 + \left(\frac{u(b)}{b}\right)^2 + 3\left(\frac{u(h)}{h}\right)^2 + u(L)\left[\left(\frac{1}{L - L_0}\right)^2 + 4\left(\frac{3L + 2L_0}{L^2 + (L + L_0)^2}\right)^2\right]}$$ (B.6)

where

$u(E)$ is the standard uncertainty in the measurement of elastic modulus;

E is the elastic modulus;

Δd is the change in displacement of the mid-point of the specimen corresponding to the change ΔP in the load force;

L is the gauge length of the extensometer.

B.4   Acoustic resonance

B.4.1   General

For each mode of excitation, calculate the standard uncertainty in frequency $u(f)$ from the five consecutive measurements. Calculate the standard uncertainties in the dimensions, L, b, and h, and in the mass, m, according to B.1.

B.4.2   Elastic modulus

Calculate the relative standard uncertainty in the elastic modulus using

$$\frac{u(E)}{E} = \sqrt{\left(\frac{u(f_1)}{f_1}\right)^2 + \left(\frac{u(f_2)}{f_2}\right)^2 + 3\left[\left(\frac{u(L)}{L}\right)^2 + \left(\frac{u(h)}{h}\right)^2\right] + \left(\frac{u(m)}{m}\right)^2 + \left(\frac{u(b)}{b}\right)^2}$$ (B.8)

where

$u(E)$ is the standard uncertainty in the measurement of elastic modulus;

E is the elastic modulus;

$u(f)$ is the standard uncertainty in frequency;

f is the frequency;

L is the gauge length of the extensometer.

B.4.3   Poisson ratio

Calculate the standard uncertainty in the Poisson ratio v using

$$u(v) = (1+v)\sqrt{2\left[\left(\frac{u(f_1)}{f_1}\right)^2 + \left(\frac{u(f_2)}{f_2}\right)^2 + \left(\frac{u(L)}{L}\right)^2 + \left(\frac{u(h)}{h}\right)^2\right]} \qquad (B.9)$$

where

$u(v)$ is the standard uncertainty in the Poisson ratio;

v is Poisson's ratio for the test material;

$u(f)$ is the standard uncertainty in frequency;

f is the frequency;

L is the gauge length of the extensometer;

h is the height of the specimen.

Annex C
(informative)

Measurement of Poisson ratio

C.1   Purpose

For purposes of computer-aided design, Young's modulus alone gives insufficient information for the accurate evaluation of stress concentration at interfaces between the component materials of an appliance. For complete specification of the elasticity of an isotropic material, a second parameter is required. When Young's modulus is given, the conventional choice is Poisson's ratio. Alternative parameters are the shear modulus or the bulk modulus.

C.2   Terms and definitions

C.2.1   Poisson's ratio

Poisson's ratio is the ratio of the negative transverse strain to the longitudinal strain of a material subject to a uniaxial stress. Poisson's ratio is a dimensionless quantity.

C.2.2   Transverse strain

Transverse strain is the ratio of the change in a dimension of a specimen relative to the initial dimension on application of a tensile or compressive stress normal to that dimension. Transverse strain is a dimensionless quantity.

C.2.3   Longitudinal strain

Longitudinal strain is the ratio of the change in a dimension of a specimen relative to the initial dimension on application of a tensile or compressive stress parallel to that dimension. Longitudinal strain is a dimensionless quantity.

C.2.4   Uniaxial stress

Uniaxial stress is the ratio of the equal forces applied in opposing directions along the axis of symmetry of a cylindrical body to the cross-sectional area of the body normal to the axis. The units are pascal, denoted as Pa.

C.3   Measurement methods

C.3.1   Tensile strain method

C.3.1.1   Specimen preparation

When specimens of sufficient size can be manufactured to determine strength according to ISO 6892-1, they may be used for the measurement of elastic modulus and Poisson's ratio.

C.3.1.2   Apparatus

As given in 8.6.1.1 with the addition of the following:

C.3.1.2.1 **Extensometer**, capable of measuring and recording a change in transverse dimension to 1 μm simultaneously with the independent measurement of longitudinal dimension.

C.3.2 Flexural method in three-point bending mode

C.3.2.1 Specimen preparation

Once measurements have been completed as described in 8.3, cut the platelets parallel to the long edges to form three rectangular specimens of breadth b = (6,0 ± 0,5) mm, (3,0 ± 0,5) mm and (1,5 ± 0,5) mm. Remove excess metal from the cut edges so that there is no protrusion above broad surfaces.

C.3.2.2 Measurement

Measure the breadth of each specimen at five positions along the section of the plate that will lie between the supports and find the average breadth b. Evaluate the ratios b/h for each specimen and the function $f(b/h)$ for each specimen using the values in Table 4.

Repeat the procedure described in 8.6 for each specimen and evaluate the apparent elastic modulus E_a according to Table 3. Evaluate the experimental uncertainty $\delta i(E_a)$ according to Annex B.

C.3.2.3 Non-linear least squares estimation of the elastic modulus and Poisson's ratio

Calculate the difference between the calculated and measured value of the apparent elastic modulus for each specimen i by the formula and weighted by the inverse of the standard variance $u(E_{a,i})^2$ in the apparent elastic modulus

$$\Delta E_{a,i} = \frac{1}{u\left(E_{a,i}\right)^2}\left(E_{a,i} - \frac{E}{\left(1 - f_i \nu^2\right)}\right) \tag{C.1}$$

f_i is the factor given in Table 4, appropriate to the ratio of specimen breadth to depth.

This can be approximated by a linear expression in the corrections (denoted by Δ) to the parameters E and ν^2

$$\Delta E_{a,i} = \frac{1}{u(E_{a,i})^2}\left[\left(1 + f_i \nu^2\right)\Delta E + E f_i \left(1 + 2 f_i \nu^2\right)\right] \tag{C.2}$$

Starting from a value of E equal to the least measured value of E_a, and a value of ν = 0,34, apply the linear least squares method to minimise the mean squared difference between these expressions in order to obtain estimates for the corrections ΔE and $\Delta \nu^2$. Adjust the parameters E and ν^2 by these amounts and repeat. Two repetitions give convergence; i.e. the corrections become small. Evaluate the variances and co-variances for E and ν and report 95% confidence limits.

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