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Dentistry — Metallic materials for fixed and removable restorations and appliances

Art dentaire — Matériaux métalliques pour les restaurations fixes et amovibles et les appareillages

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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

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

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

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

ISO 22674 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This first edition cancels and replaces the following composition-derived International Standards: ISO 1562, ISO 6871-1, ISO 6871-2, ISO 8891 and ISO 16744.

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 should 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.

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 Subcommittee 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 appliances and restorations, including metallic materials recommended for use either with or without a ceramic veneer, or recommended for both uses, and specifies their requirements. It further specifies requirements with respect to packaging and marking the products and to the instructions to be supplied for the use of these materials.

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., wire, bracket, band and screw).

2 Normative references

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

ISO 1942, *Dentistry — Vocabulary*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 6892, *Metallic materials — Tensile testing at ambient temperature*

ISO 7500-1:2004, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

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

ISO 9693, *Metal-ceramic dental restorative systems*

ISO 10271:2001, *Dental metallic materials — Corrosion test methods*

ISO 15223:2000, *Medical devices — Symbols to be used with medical device labels, labelling and information to be supplied*

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 and silver

3.2
hazardous element
element that is known for its potential to produce an adverse biological effect

NOTE 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 harmful.

3.3
metallic material
material having the properties that are associated with an alloy, noble metal or base metal

NOTE This may be a pure element, commercially pure metal or an alloy.

3.4
casting alloy
metallic material designed for casting into a dental investment mould

3.5
ceramic veneer
thin ceramic surface layer present on a metallic material restoration to provide an aesthetic effect

3.6
metal-ceramic
dental restoration in which a ceramic veneer is bonded to a metallic material substructure by firing

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

NOTE 2 If recommended, such a metallic material may be used without a ceramic veneer.

3.7
metallic base
noble metal or base metal with highest concentration by mass fraction in the alloy

NOTE 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.

3.8
base-metal alloy
alloy having a base metal as the principal element

3.9
as-cast condition
metallurgical condition of the metallic material in its solid state after removal from the casting machine

NOTE This condition is dependent upon the manufacturer’s recommended cooling procedure (e.g., bench cooling).

3.10
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

3.11
hardening
heat-treatment producing a condition which provides a higher 0,2 % proof strength than the “as-cast” state

NOTE If recommended by the manufacturer, explicit instructions should be given in the instructions for use.

3.12
softening
heat-treatment producing a condition which provides a lower 0,2 % proof strength than the “as-cast” state

NOTE If recommended by the manufacturer, explicit instructions should be given in the instructions for use.

3.13
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

3.14
veneer
thin covering of surface material applied to a coarser base material

4 Classification

For the purposes of this International Standard a metallic material is classified, according to its mechanical properties as 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 restorations, 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:** intended for low stress bearing single-tooth fixed restorations, e.g. veneered or unveneered one-surface inlays, veneered crowns.

— **Type 2:** intended for single tooth fixed restorations, e.g. crowns or inlays without restriction on the number of surfaces.

— **Type 3:** intended for multiple unit fixed restorations, e.g. bridges.

— **Type 4:** intended for appliances with thin sections that are subject to very high forces, e.g. removable partial dentures, clasps, thin veneered crowns, wide-span bridges or bridges with small cross-sections, bars, attachments, implant retained superstructures.

— **Type 5:** intended for appliances in which parts require the combination of high stiffness and strength, e.g. thin removable partial dentures, parts with thin cross-sections, clasps.

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 [see 9.1 a)] either by name or symbol.

5.1.2 Permitted deviation from the reported composition for elements

For silver-based or noble-metal alloys, the percentage of each of the constituents of the alloy shall not deviate by more than 0,5 % (mass fraction) from the values stated on the package label or insert [see 9.1 a)].

For base-metal alloys, all elements present in excess of 20 % (mass fraction) shall not deviate by more than 2,0 % (mass fraction) from the value stated on the package or label or insert. Those present in excess of 1,0 % (mass fraction) but not in excess of 20 % (mass fraction) shall not deviate by more than 1,0 % (mass fraction) from the value stated on the package or label or insert [see 9.1 a)].

5.2 Hazardous elements

5.2.1 Recognized hazardous elements

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

5.2.2 Permitted limits for the hazardous elements cadmium and beryllium

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

5.2.3 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).

5.3 Biocompatibility

See the introduction for guidance on biocompatibility.

5.4 Mechanical properties

5.4.1 General

Testing shall be done according to 8.3.2.

The requirements in Table 1 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 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 may be classified differently (according to Table 1) 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 1 — Mechanical properties

Type	Proof strength of 0,2 % non-proportional extension $R_{p0,2}$ MPa minimum	Elongation after fracture % minimum	Young's modulus GPa minimum
0	—	—	—
1	80	18	—
2	180	10	—
3	270	5	—
4	360	2	—
5	500	2	150

5.4.2 Proof strength 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 strength 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 strength of 0,2 % non-proportional extension, the metallic material fails to comply with the requirement.

NOTE 1 Two sets of six specimens are produced (see 7.3.1). One of these sets 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 set of six specimens are rejected on the basis of 8.3.2 and replaced with specimens from the second set, all twelve specimens shall be tested. If at least eight results meet the requirement for proof strength 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 strength 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 strength of 0,2 % non-proportional extension, the metallic material complies with the requirement.

NOTE 3 Under this compliance criterion, if one specimen from the first set 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. N.B. Only one replacement is possible for a borderline metallic material.

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

5.4.2.2 Mean value

The mean value for the proof strength of 0,2 % non-proportional extension 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, which meet the requirement for proof strength 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 sets of six specimens are produced (see 7.3.1). One of these sets is tested in the first test series. If required, replacement specimens are drawn from the second set and used in the first test series. The remaining specimens in the second set 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 set of six specimens are rejected on the basis of 8.3.2 and replaced with specimens from the second set, all twelve specimens shall be tested. If at least eight results of the specimens that meet the requirement for proof strength of 0,2 % non-proportional extension also meet the requirement for elongation to 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, which meet the requirement for proof strength 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 strength 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 strength of 0,2 % non-proportional extension, the metallic material fails to comply with requirement for elongation after fracture.

NOTE 3 Under this compliance criterion, if one specimen in the first set has been rejected on the basis of inspection after fracture (8.3.2) during the first test series and a specimen from the second set of six used as its replacement, then all five remaining specimens in the second series shall meet the requirement. N.B. Only one replacement is possible for a borderline metallic material.

If three of the results for the 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 requirements for elongation after fracture, the metallic material fails to comply with the requirement for elongation after fracture.

5.4.3.2 Mean reported value

The mean value of percentage elongation after fracture shall 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)]

5.4.4 Young's modulus

5.4.4.1 The determination of compliance with the requirement for Type 5 materials

Refer to classification Type stated in 9.1 b).

If the results for the four, five or six of the set of six specimens in the first series of tests, which meet the requirement for proof strength of 0,2 % non-proportional extension and the percentage elongation after fracture, also meet the requirement for Young's modulus, the metallic material complies with the requirement for Young's modulus.

If two or fewer of the results for the first set of six specimens in the first series of tests, which meet the requirement for proof strength of 0,2 % non-proportional extension and the percentage elongation after fracture, meet the requirement for Young's modulus, the metallic material fails to comply with the requirement for Young's modulus.

NOTE 1 Two lots of six specimens are produced (see 7.3.1). 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 for the set is reached when the number of specimens tested in the first test series 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 twelve specimens shall be tested. If at least eight results of the specimens, that meet the requirement for proof strength of 0,2 % non-proportional extension and the requirement for elongation fracture, also meet the requirement for Young's modulus, the metallic material complies with the requirement.

If three of the results for the first set of six specimens in the first series of tests that meet the requirement for proof strength of 0,2 % non-proportional extension and the percentage elongation after fracture have values of Young's modulus which meet the requirement, all remaining specimens shall be tested in a second series of tests. If five or six of the results from these remaining specimens have values for Young's modulus, proof strength of 0,2 % non-proportional extension and the percentage elongation after fracture which meet the requirements, the metallic material complies with the requirement for Young's modulus. Otherwise, the metallic material fails to comply with the requirement for Young's modulus.

NOTE 3 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 used as its replacement, then all five remaining specimens in the second lot shall meet the requirement. N.B. Only one replacement is possible for a borderline metallic material.

5.4.4.2 Mean value

The mean value of the Young's modulus shall not differ by more than ± 10 % of the value stated in the literature accompanying the package [see 9.1 e)].

5.5 Density

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)].

5.6 Corrosion resistance for material integrity

The total metal ion release from the metallic material into the specified solution (see 8.5.4) at (37 ± 1) °C in a time period of (7,0 ± 0,1) d shall not exceed 200 µg cm^{-2} when tested according to 8.5.

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.

5.7 Tarnish resistance

This requirement shall apply only if a claim is made in the “Information” in the literature that accompanies the package that the product is “tarnish-resistant”.

After exposure to the specified tarnishing environment (specified in 8.6), 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.

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

For an alloy with a solidus temperature less than 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)].

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)].

For a commercially pure metal the melting point shall not differ 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.

5.9 Thermal expansion coefficient

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

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)].

NOTE The measured value for the coefficient of linear expansion is compared with the manufacturer’s value for quality control. Compliance with this requirement cannot provide an assurance that the alloy and any particular ceramic are compatible.

5.10 Information, instructions and marking

All the requirements specified in Clauses 9 and 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 7.3 and shall be from one lot. Further samples and packaging materials shall be made available for inspection in accordance with 8.1.

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

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 heat treatment is recommended in the manufacturer's instructions for use

If 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 each of the oxidation and ceramic firing simulations.

If the product is also recommended for use without application of a ceramic veneer, 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 casting metallic materials the appropriate condition is the bench-cooled state.

7.3 Specimens

7.3.1 Mechanical properties

7.3.1.1 Metallic materials for which production of conventional specimens is possible

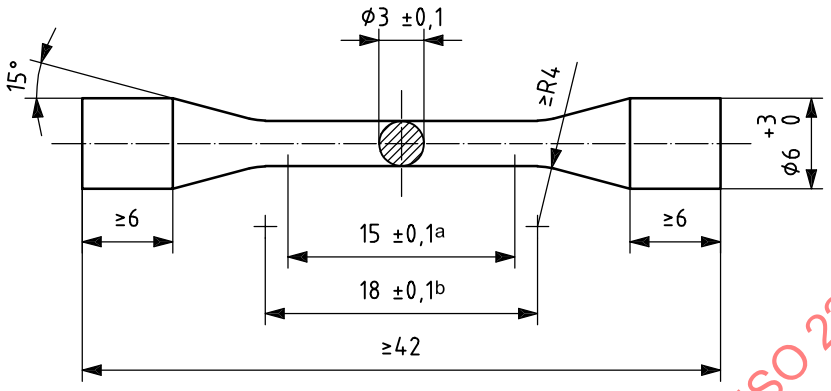
Prepare two lots of six specimens that comply with Figure 1 or Figure 2, formed, finished and heat-treated in accordance with 7.1 and 7.2.

If the product is intended for use both with and without a ceramic veneer, four sets 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

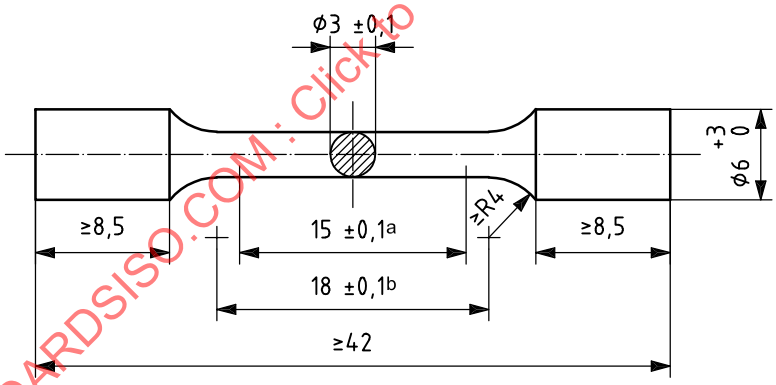


a Gauge length.

b Parallel section of test specimen.

Figure 1 — Test specimen with conical shoulders

Dimensions in millimetres



a Gauge length.

b Parallel section of test specimen.

Figure 2 — Test specimen with radial shoulders

7.3.1.2 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 Figures 1 or 2.

7.3.2 Density measurement

Use material taken from retail packages, without further processing, provided it be free from pores.

Otherwise, process the material according to the manufacturer's instructions for use, given in 9.2, if possible to a regular shape and geometry for the test specimens (e.g., rod). With irregularly shaped material choose pieces on which all parts of the external surfaces are visible.

7.3.3 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).

7.3.4 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.

7.3.5 Tarnish resistance

To test for tarnish resistance in accordance with 8.6, prepare two disc specimens with dimensions of (10 ± 1) mm diameter × (0,5 ± 0,1) mm thickness, 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, 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).

8 Test methods

8.1 Information, instructions and marking

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

8.2 Chemical composition

Analyse quantitatively for all 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 0,1 % (mass fraction) and to 0,01 % (mass fraction) 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.

Analyse quantitatively for beryllium, cadmium 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 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 any element that is not a declared element [see 9.1 a)] or a hazardous element is detected and present in a concentration greater than or equal to 1,0 % (mass fraction), 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 properties

8.3.1 Apparatus

8.3.1.1 **Micrometer**, accurate to 0,01 mm with a measuring capacity of 25 mm.

8.3.1.2 **Mechanical testing machine**, of capacity up to 10 kN (for both frame and load cell), with an accuracy greater 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**, compatible with the specimen gauge length. Accurate to greater than 1 µm.

8.3.1.4 **Travelling microscope**, with at least 10 × magnification, accurate to 0,01 mm with a measuring capacity of 25 mm.

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. (An alternative instrument for measuring the gauge length may be used in place of the travelling microscope provided it have an accuracy of 0,02 mm or better).

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^{-1} 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. A more accurate method, such as one using a laser extensometer, can also be used to determine the percentage elongation.

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 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 from the second lot of specimens and repeat the test.

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

Test no more than twelve specimens.

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

8.3.3.1 Calculation of the proof strength of 0,2 % non-proportional extension

For each specimen, using the force-extension diagram, determine the force at which there is 0,2 % non-proportional extension and calculate the proof strength, $R_{p0,2}$, by dividing this force by the original cross-sectional area within the gauge length of the test piece. This property may also be obtained without plotting the force extension curve, by using an automatic device (e.g., a microprocessor).

Determine the proof strength 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 proof strength of 0,2 % 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 Calculation of the mean value for proof strength of 0,2 % non-proportional extension

Calculate the proof strength 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 of the percentage elongation after fracture

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

Calculate the elongation after fracture by taking the difference between the gauge length readings before and after testing as a 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 Calculation of the mean value for the percentage elongation after fracture

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 %.

8.3.5 Young's modulus

8.3.5.1 Calculation of Young's modulus

Determine Young's modulus using the extensometer recording for extension and the testing machine recording for the force.

Report the Young's modulus for all specimens that were tested and not rejected following the post-test inspection. Report the results to the nearest 1 GPa.

8.3.5.2 Calculation of the mean value for Young's modulus

Calculate the Young's modulus as the mean of the values of all tested specimens tested and not rejected following the post-test inspection. Report the value to the nearest 5 GPa.

8.4 Density

8.4.1 Preparation of test specimens

Determine the density of the metallic material, prepared according to 7.3.2.

8.4.2 Reagents

8.4.2.1 **Water**, of grade 3 in accordance with ISO 3696:1987.

8.4.2.2 **Ethanol or methanol**, analytical grade.

8.4.3 Apparatus

8.4.3.1 Balance, pycnometer or other suitable device, having an opening and volume appropriate for the specimens to be used.

8.4.4 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 (8.4.2.2) and clean for 2 min in an ultrasonic bath. Rinse the specimens in water (8.4.2.1) 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, 8.4.3.1) 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}.

8.5 Corrosion resistance

8.5.1 Preparation of test specimens

Use specimens produced in accordance with 7.3.3.

8.5.2 Reagents

Use reagents according to 4.1.3 in ISO 10271:2001.

8.5.3 Apparatus

Use apparatus according to 4.1.4 in ISO 10271:2001. The sensitivity of the water-pH meter should be ± 0,05 pH units.

8.5.4 Test solution

Prepare a fresh solution for each test according to 4.1.5 in ISO 10271:2001.

8.5.5 Test procedure

Determine the surface area of each test specimen or sample (7.3.3) to the nearest 0,1 cm^2. Immerse the specimens in ethanol or methanol (8.5.2) and clean for 2 min in an ultrasonic bath. Rinse the specimens in water and dry them with water-free and oil-free compressed air. Now, follow the test procedure according to 4.1.7 in ISO 10271:2001.

In parallel with the production of sample solutions, produce a reference sample 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 (7,0 ± 0,1) d.

8.5.6 Analysis

By using an instrumental analytical method of adequate sensitivity (e.g., 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).

8.5.7 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 (7.3.3). Report the value for each of elements in the units "µg cm^{-2} in 7 days", rounded to the nearest one 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.

8.6 Tarnish resistance (sodium sulfide test)

8.6.1 Reagents and equipment

8.6.1.1 **Sodium sulfide hydrate**, (approximately 35 % Na_2S), $Na_2S{\cdot}(7\text{-}9)H_2O$, analytical grade.

8.6.1.2 **Water,** grade 2 according to ISO 3696:1987.

8.6.1.3 **Dipping device**, that dips the test specimen into the test solution for 10 s to 15 s once every minute.

8.6.1.4 **Ethanol**, analytical grade.

8.6.2 Test solution

Prepare an aqueous solution of 0,1 mol/litre sodium sulfide immediately before use. Dissolve 22,3 g sodium sulfide hydrate (8.6.1.1) in water (8.6.1.2) and dilute to 1 000 ml with water.

8.6.3 Preparation of test specimens

Use specimens produced in accordance with 7.3.5. Cold mount them separately in clear resin. Wet grind and polish using standard metallographic procedures, finishing with 1 µm water-based polishing paste. Use fresh grinding paper and polishing paste for the preparation of specimens from different alloys.

Immerse in ethyl alcohol (ethanol) (8.6.1.4) and clean for 2 min in an ultrasonic bath. Rinse in water (8.6.1.2), and dry with oil-free and water-free compressed air.

8.6.4 Procedure

Take one of the specimens, prepared in accordance with 8.6.3 and fix it in the dipping device. Use 1 000 ml of solution and maintain its temperature at (23 ± 2) °C. Dip the specimen into the solution for 10 s to 15 s once every minute over a period of (72 ± 1) h. Replace the test solution after (24 ± 1) h and again after (48 ± 1) h. After (72 ± 1) h remove the test specimen from the dipping device. Rinse the specimen thoroughly in water (8.6.1.2), dip in ethanol (8.6.1.4) and then dry with a jet of water-free and oil-free compressed air.

CAUTION — This test should be carried out in a properly operating fume cupboard.

8.6.5 Inspection

Inspect using eyesight with nominally normal trichromatic vision without magnification. Corrective (non-magnifying) non-tinted lenses may be worn. Inspect under white light that has an intensity of at least 1 000 lx and at a distance not exceeding 25 cm. Examine the surface of the specimen that had been dipped into the solution and compare this with the surface of the specimen that had not been dipped. Brush or rub the surface gently with a soft brush or cloth to assess the ease with which the products of tarnish may be removed.

8.6.6 Treatment of results

Record and report any visible differences in colour and reflectivity, which have resulted from exposure to the solution. Record the ease with which the products of tarnish may be removed. If there is no change for either colour or reflectivity or both of these, this is to be reported.

If there is a second set of specimens that has been subjected to a different heat treatment repeat this test and record separately.

Classify the result according to 5.7.

8.7 Solidus and liquidus temperatures (alloys) or melting point (commercially pure metals)

By using the cooling curve method, determine the solidus and liquidus temperatures for an alloy or melting point for commercially pure metal to an accuracy of ± 10 °C for metallic materials with solidus or melting point temperatures below or equal to 1 200 °C, and ± 25 °C for metallic materials with solidus or melting point temperatures above 1 200 °C.

Other standard procedures that have equivalent accuracy may be used.

8.8 Linear thermal expansion

Use specimens of the type described in 7.3.4.

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 for each specimen from the linear expansion/temperature recordings.

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

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; each element that is present as an element in a mass fraction greater than 0,1 % shall be declared and 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 thus:

   — cobalt-based dental casting 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 proof strength of 0,2 % non-proportional extension;

d) the percentage elongation after fracture;

e) Young´s modulus;

f) the density;

g) if appropriate, a statement that the product is tarnish resistant (see 5.7);

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 or as a metal-ceramic 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”.

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:

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 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) this requirement is not applicable. Instead the information shall be printed on the packaging which is in direct contact with the metallic material.

10.2 Labelling on the package

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

a) manufacturer's or distributor'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 shall be identical to that reported in 9.1 b);

d) lot number;

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

f) if the metallic material contains more than 0,1 % (mass fraction) nickel, a warning symbol (a triangle within which there is an exclamation mark), in accordance with 3.4 of ISO 15223:2000;

g) 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.