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Dentistry — Brazing materials

Art dentaire — Produits pour brasage

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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 9333 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This second edition cancels and replaces the first edition (ISO 9333:1990), subclauses 4.1 and 4.4 of which have been technically revised:

- a) in 4.1 by the introduction of upper limits for cadmium, beryllium and lead;
- b) in 4.4 by a reduction in the mechanical strength of a brazed joint;
- c) further, the requirement for flow temperature has been cancelled.

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Dentistry — Brazing materials

1 Scope

This International Standard specifies requirements and test methods for brazing materials suitable for use in metallic restorations.

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

ISO 7405, *Dentistry — Preclinical evaluation of biocompatibility of medical devices used in dentistry — Test methods for dental materials*

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

ISO 10993-1, *Biological evaluation of medical devices — Part 1: Evaluation and testing*

3 Terms and definitions

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

3.1

brazing material

alloy suitable for use as a filler material in operations in which dental alloy parts are joined by brazing to form dental appliances and restorations

3.2

noble metal

gold, platinum, palladium, ruthenium, iridium, osmium and rhodium

3.3

base metal

all metallic elements with the exception of noble metals and silver

4 Requirements

4.1 Chemical composition

4.1.1 Reported composition

For all elements that are present in excess of 1,0 % (mass fraction), the percentage by mass of each of the constituent elements shall be declared by the manufacturer and shall be reported as instructed in 8 c) to a precision of 0,1 % (mass fraction). Any element that is present in a concentration in excess of 0,1 % (mass fraction), but not in excess of 1,0 % (mass fraction), shall be identified as instructed in 8 c) either by name or symbol.

4.1.2 Permitted deviation from the reported composition

For silver-based or noble-metal-based brazing materials the percentage of each of the constituents shall not deviate by more than 0,5 % (mass fraction) from the values stated in the manufacturer's or distributor's instructions for use [see 8 c)].

For base-metal-based brazing materials, all elements present with more than 20 % (mass fraction) shall not deviate from the value stated in the manufacturer's or distributor's instructions for use by more than 2 % (mass fraction). Those present in excess of 1 % (mass fraction) but not in excess of 20 % (mass fraction) shall not deviate from the value stated in the manufacturer's or distributor's instructions for use by more than 1 % (mass fraction) [see 8 c)].

4.1.3 Hazardous elements

4.1.3.1 Recognised hazardous elements

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

4.1.3.2 Permitted limits for hazardous elements

The brazing material shall not contain more than 0,02 % (mass fraction) of cadmium or beryllium or lead. If the brazing material contains more than 0,1 % (mass fraction) of nickel, the percentage shall not exceed the amount indicated on the package [see 9.2 e)] or insert [see 8 d)].

4.2 Biocompatibility

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.

4.3 Corrosion resistance

A comparison of the surface of the tested specimens with that of the untested specimens shall not reveal any visible evidence that a chemical reaction has occurred.

Testing shall be carried out in accordance with 7.3.

4.4 Mechanical strength (tensile strength) of brazed joint

The tensile strength shall exceed 250 MPa. If the 0,2 % proof strength of either one or both of the metallic materials to be joined by the brazing material is below 250 MPa, the tensile strength shall exceed the lower of the two.

Testing shall be carried out in accordance with 7.4.

4.5 Melting range

The solidus temperature and the liquidus temperature of the brazing material shall be within ± 20 °C of the values given in the manufacturer's instructions [see 8 e)].

Testing shall be carried out in accordance with 7.5.

5 Sampling

The amount of test material shall be sufficient to prepare the specimens required in 6.2 and 6.3, including provision for a second set for tensile testing.

6 Preparation of test specimens

6.1 General

The test specimens consist of the recommended metallic materials joined with the brazing material according to the manufacturer's instructions. Prepare the test specimens by the "lost wax process" of investment casting. Methods other than casting may be recommended by the manufacturer for the metallic material to be tested with brazing materials. Use such a method, if recommended by the manufacturer. Follow the manufacturer's instructions relating to the processing of the metallic material(s) and the brazing material including necessary aids, casting and brazing equipment as given in 8 f) and 8 g).

6.2 Specimens for corrosion testing

Prepare four pieces of dimensions approximately 10 mm \times 10 mm \times 1 mm and clean the surfaces. Cut the samples in half (5 mm \times 10 mm \times 1 mm). Use two halves of the pieces for brazing. If two different metallic materials are to be brazed, use one of each for the two halves. Set the gap between the cut pieces at (0,2 \pm 0,1) mm. Use the recommended flux and follow the manufacturer's instructions on brazing. Following brazing remove at least 0,1 mm from all surfaces of the samples using standard metallographic procedures ending with wet silicon carbide paper of grade P 1200 in accordance with ISO 6344-1. Use the same grinding paper solely for the preparation of specimens of the same composition.

6.3 Specimens for tensile testing

Prepare a set of six specimens of the metallic material(s) to be brazed, which comply with either Figure 1 or Figure 2. Replace specimens that have visible shrinkage, defects or porosities. Cut the specimens of the set at right angles to its long axis at the midpoint of the gauge length using a fine saw. Remove rough edges. Grind the cut surface smooth, plane and perpendicular to the axis of the test specimen. Support the two halves of the specimens and align them in an investment or a rigid jig. If two different metallic materials are to be brazed, use one of each for the two halves. Set the gap between the cut pieces at (0,2 \pm 0,1) mm. Use the recommended flux and follow the manufacturer's instructions on brazing.

After brazing, ensure that the diameter of each tensile specimen is within the tolerances given in Figure 1 or Figure 2, and does not show visual evidence of radial run-out when rotated.

If the proof strength of non-proportional elongation of the dental metallic material for which the brazing material is recommended is less than 250 MPa, prepare an additional set of six specimens of the metallic material to be brazed to determine its 0,2 % proof strength.

Dimensions in millimetres

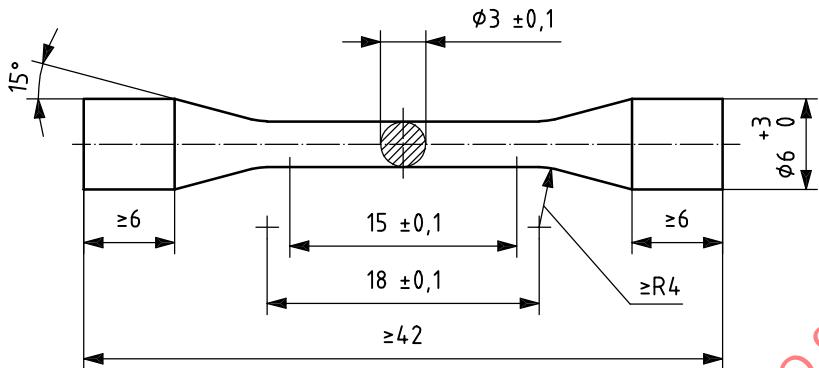


Figure 1 — Test specimen with conical shoulders

Dimensions in millimetres

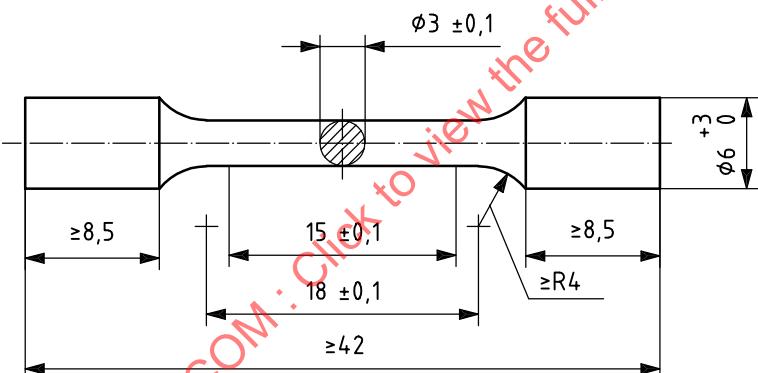


Figure 2 — Test specimen with radial shoulders

7 Testing

7.1 Visual inspection

7.1.1 Specimens

Without the aid of magnification, inspect the surface of specimens to be tested for visible defects. Replace any test specimens that have defects.

7.1.2 Information and instructions, packaging and labelling

Visually inspect to check that the requirements specified in Clauses 8 and 9 have been met.

7.2 Chemical composition

Determine the composition using analytical procedures with sensitivities appropriate to the concentration of each element and its permitted deviation from the stated value or permitted limit.

7.3 Corrosion testing

Perform the static immersion test with four of the brazed specimens (6.2), using the test solution and test method in accordance with ISO 10271:2001, 4.1.

After the test, inspect the brazed gap of the specimens under a microscope at a magnification of $\times 10$.

7.4 Tensile testing

7.4.1 Apparatus

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

7.4.1.2 Universal mechanical testing machine, with a capacity in excess of 4 kN for both frame and load cell, and that operates at a cross-head speed up to $2 \text{ mm} \cdot \text{min}^{-1}$.

Select grips appropriate for the specimens to be used. Conduct tensile testing with a system that has a spring constant (defined as the ratio of the load applied to its elastic deflection) very significantly greater than the load-extension ratio of the specimen under test. (The system comprises the testing machine frame, load cell and attachments). Record the specimen extension free from the machine deflection.

7.4.2 Test procedure

Determine the tensile strength on a set of six brazed specimens, produced in accordance with 6.3.

If applicable, determine the 0,2 % proof strength of non-proportional elongation for the metallic material intended for brazing. Use a set of six specimens.

Measure the diameter of each specimen to an accuracy of 0,01 mm using the micrometer (7.4.1.1).

Conduct the test at ambient room temperature.

Load the specimens in tension into the universal mechanical testing machine (7.4.1.2) at a cross-head speed of $(1,5 \pm 0,5) \text{ mm} \cdot \text{min}^{-1}$ up to the fracture point of the specimens. Record the force and elongation continuously.

Examine the fractured specimen to determine whether the fracture occurred within the gauge length of the specimen. If the fracture has taken place outside the gauge length, reject the specimen and its result. Replace the specimen and repeat the test.

Calculate the tensile strength of each specimen on the basis of the original cross-sectional area, using the force derived from the force/extension recording.

If appropriate, determine the force at which there is 0,2 % non-proportional extension, from the force/extension records of the metallic material and calculate the proof strength of non-proportional elongation on the basis of the original cross-sectional area.

7.4.3 Evaluation of the tensile test results

If four, five or six brazed specimens are found to exceed the minimum requirement given in 4.4, the brazing material satisfies the tensile strength requirement of this International Standard.

If two or fewer brazed specimens are found to comply with the minimum requirement given in 4.4, the brazing material fails the tensile strength requirement of this International Standard.

If three brazed specimens are found to comply with the minimum requirement given in 4.4, produce a second set of six test specimens and repeat the test.

If, in the second test set, five or six test specimens are found to exceed the minimum requirement given in 4.4, then the brazing material satisfies the tensile strength requirement of this International Standard.

7.5 Melting range

Determine the melting range by the cooling curve method or other methods of equivalent accuracy. By using the cooling curve method, determine the solidus and liquidus temperatures to an accuracy of $\pm 10^{\circ}\text{C}$.

Report to the nearest 10°C .

8 Information and instructions

The manufacturer's or distributor's information and instructions for use shall contain at least the following information about applications, chemical and physical properties and processing of the brazing material:

- a) manufacturer's or distributor's name or trademark and address;
- b) designation or trade name of the brazing material;
- c) the composition of the brazing material; all elements present in concentrations greater than 0,1 % (mass fraction) shall be declared and all elements present in concentration greater than 1,0 % (mass fraction) shall have their concentrations given;
- d) if the dental brazing material contains more than 0,1 % (mass fraction) nickel, a warning and the percentage (mass fraction) in which it is present shall be given; adequate detailed instructions regarding safety precautions shall be given in the package or in the accompanying literature;
- e) melting range (solidus and liquidus temperature) in degrees centigrade;
- f) recommendation for fluxes and for metallic materials and/or combinations;
- g) detailed instructions for brazing recommended metallic materials.

9 Marking and labelling

9.1 Marking

The brazing material, or the primary pack or any other form in which the brazing material is supplied, shall be clearly marked to identify the manufacturer or distributor and the brazing material.

9.2 Package

The brazing material shall be packed in accordance with accepted commercial practice to prevent any contamination or damage.

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

- a) manufacturer's or distributor's name or trademark and address;
- b) designation or trade name of the brazing material;
- c) lot number;
- d) minimum net mass, in grams;
- e) a warning, if the dental brazing material contains nickel [see 8 d)] and the percentage (mass fraction) in which it is present;
- f) intended use.

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