INTERNATIONAL STANDARD

ISO 16402

First edition
2008-05-15

Implants for surgery — Acrylic resin cement — Flexural fatigue testing of acrylic resin cements used in orthopaedics

Implants chirurgicaux — Ciment à base de résine acrylique — Essais de fatigue flexurale des ciments à base de résine acrylique utilisés en orthopédie

Reference number
ISO 16402:2008(E)

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

Published in Switzerland

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 16402 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

Implants for surgery — Acrylic resin cement — Flexural fatigue testing of acrylic resin cements used in orthopaedics

1 Scope

This International Standard applies to resin cements based on poly(methacrylic acid esters) and specifies the procedure for determining the fatigue behaviour of the polymerized cement.

This International Standard does not cover the hazards associated with the use of the cement in respect of either the patient or the user of the cement.

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 5833:2002, *Implants for surgery — Acrylic resin cements*

ISO 16428, *Implants for surgery — Test solutions and environmental conditions for static and dynamic corrosion tests on implantable materials and medical devices*

3 Test method

The tests make use of rectangular bar-shaped test specimens. The method for preparing these specimens is described in Clause 5.

The bending strength of the resulting test specimens is determined by means of a four-point bend test under quasi static and cyclic loading conditions. Under cyclic loading conditions two test methods are described. The first one follows the Wöhler method (S/N curve method) and determines the behaviour over the full range of stress levels. The second one (low stress method) determines the behaviour at lower stress levels only. These lower stress levels, which are specified, are thought to correspond to stress levels expected to be encountered in clinical use.

4 Apparatus

4.1 Equipment for mixing cement, as recommended by the cement manufacturer.

4.2 Moulds, made of a suitable material to produce rectangular bar-shaped specimens directly in their final shape with the dimensions of 75 mm length, 10 mm width and 3,3 mm depth. If preferred, one or more moulds to produce plates of 3,3 mm thickness may be used and the specimens subsequently cut to size using a saw. All surfaces of the moulds which come into contact with the cement shall be finely finished with 400 grade emery paper.

NOTE Materials with sufficient stiffness, as for example aluminium alloys or stainless steel, have been found to be suitable.

4.3 Flat, smooth plates, (two for each mould) of a suitable material and size to cover the upper and lower surfaces of the moulds (4.2).

4.4 Mould release agent, to facilitate separation of the specimens from the moulds.

NOTE Teflon-spray, vacuum grease or beeswax have been found to be suitable.

4.5 Polyester film, to cover the plates of the mould.

4.6 C-clamp(s) or **other device(s)**, for clamping the mould(s) between the top and bottom plates.

4.7 Bend test machine, capable of applying loads up to 200 N which optionally can increase linearly or can cycle sinusoidally over a range of loads between 0 N and the selected value at a frequency of 5 Hz, and which is equipped with a device for measuring, controlling and recording the load to an accuracy of $\pm$ 2 N.

4.8 Four-point bend test rig, having the dimensions shown in Figure 1 (corresponding to ISO 5833:2002, Annex F) with means of preventing initial misalignment, changes of alignment and walking-off of the test specimen on the supports during the test. The loading points should be of the rolling type and have a diameter of 3 mm. The test rig should be such that equal loads are applied to all loading points.

4.9 Water bath, to maintain the test specimen and the loading fixtures at a temperature of (37 ± 1) °C.

4.10 Saw.

4.11 Emery paper, 400 grade.

5 Preparation of test specimens

5.1 General conditions

The mould(s) (4.2), plates (4.3), mixing equipment (4.1) and the cement to be used (both powder and liquid components) shall be maintained at (23 ± 2) °C for at least 2 h before casting the test specimen(s). The specimen(s) shall be cast at (23 ± 2) °C.

5.2 Preparation procedure

5.2.1 Cover the surfaces of the mould which come into contact with the cement, with a thin film of the mould release agent (4.4).

5.2.2 Cover the bottom plate of the mould with polyester film (4.5). Place the mould on top of the film-covered plate.

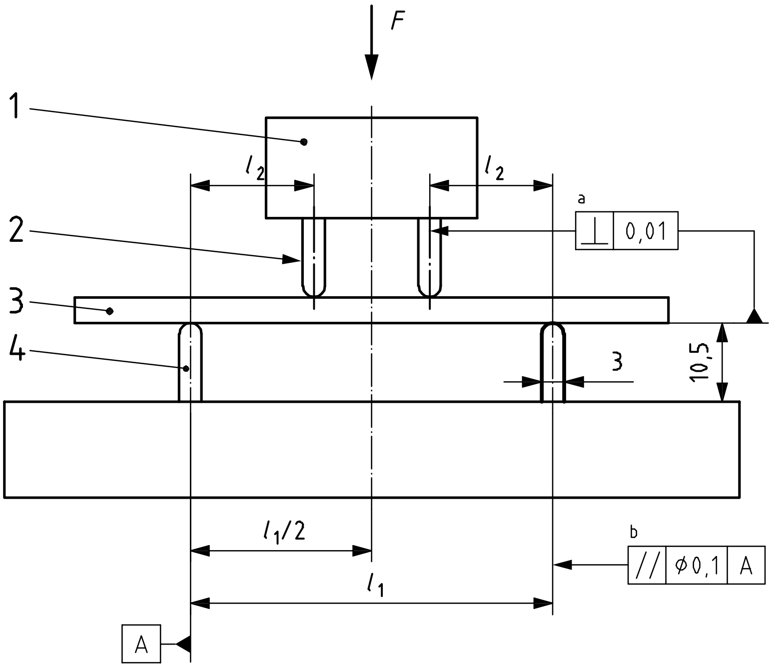
5.2.3 Mix the components of cement, following the manufacturer's instructions. Use a sufficient quantity of cement to make the required number of specimens.

NOTE A good procedure is the following: obtain a large batch of cement powder and a large batch of cement liquid from the manufacturer. Subdivide these batches into smaller units of powder and liquid, then store. Mix the number of these smaller units which is needed to make the required number of specimens. If possible, make all the specimens to be tested from a single batch of cement powder and a single batch of cement liquid. If this is not possible, use the minimum possible number of cement batches. In both cases record the batch numbers.

5.2.4 Fill the mould(s) with the cement immediately after the mixing is completed, cover with a sheet of the polyester film, add the top plate and clamp the top and bottom plates to the mould(s).

5.2.5 After approximately 1 h remove the clamp(s) (4.6), the top plate and the top polyester film and apply a mark to the top of the specimen near one end, to identify the top surface. Remove the specimen(s) from their mould(s). Wet grind the edges and the faces of the specimens which were in contact with the mould or which result from cutting with a saw (4.10), using 400 grade emery paper (4.11). The rectangular bar-shaped test specimens shall have a length of $(75 \pm 0{,}2)$ mm, width of $(10 \pm 0{,}2)$ mm, and thickness of $(3{,}3 \pm 0{,}2)$ mm.

Dimensions in millimetres



Key

1 central loading plunger
2 inner loading rollers
3 test specimen
4 outer loading rollers
F force
l_1 distance between outer loading rollers [(60 ± 1) mm]
l_2 distance between outer and inner loading rollers [(20 ± 1) mm]

NOTE See also Figure F.1 of ISO 5833:2002.

[a] All loading points.

[b] Between any two loading points.

Figure 1 — Four-point bend test rig

5.2.6 Measure the specimen thickness and width to an accuracy of $\pm$ 0,1 mm taking readings for at least three cross-sections of the specimen and calculate the mean values.

5.2.7 Examine the specimens with the naked eye. Do not exclude specimens with internal pores. Exclude specimens with discernable defects or scratches on the specimen surface.

Pores generated by the mixing procedure are unavoidable. Such pores are a feature of bone cements and influence the strength behaviour of the material. For this reason specimens with internal pores shall not be excluded. Discernable defects or scratches on the surface are not a feature of bone cements and for this reason such specimens shall be excluded.

5.2.8 Maintain the specimens in Ringer's solution according to ISO 16428 at (37 ± 1) °C for 4 weeks prior to the beginning of the test series.

6 Test procedure

6.1 Environmental conditions

Fill the water bath with Ringer's solution. Place the test rig and the test specimen in the water bath and leave until the temperature is (37 ± 1) °C. The test specimen shall be placed symmetrically in the rig so that the surface identified as “top” in the casting procedure is subjected to compressive stress.

6.2 Quasi static testing

Using the bend test machine (4.7), the four-point bend test rig (4.8) and the water bath (4.9), increase the machine test load at a uniform loading rate of (90 ± 10) N/min until fracture occurs. Record the load at fracture to an accuracy of $\pm$ 2 N. If large pores (diameter $\geqslant$ 1 mm) are found on the fracture surface repeat the test using an additional specimen. Repeat the tests until results are obtained from five specimens which do not have large pores on the fracture surface. Test results shall be reported for all specimens (including those with large pores found on the fracture surface).

6.3 Calculation and expression of the “quasi static” bending strength

For each test specimen, calculate the bending strength, σ_B, in megapascals, from the expression:

$$\sigma_\mathrm{B} = \frac{3Fa}{bh^2}$$

where

F is the force at fracture, in newtons (also referred to as the load);

b is the mean measured width of the specimen, in millimetres;

h is the mean measured thickness of the specimen, in millimetres;

a is the distance between the inner and outer loading rollers (20 mm).

Calculate the mean value and standard deviation of the individual bending strengths of the five test specimens, expressed in megapascals. The mean value is the “quasi static” strength.

6.4 Fatigue testing

6.4.1 General

Using the bend test machine (4.7), the four-point bend test rig (4.8) and the water bath (4.9), test specimens are cyclically loaded until fracture occurs or, if fracture does not occur, until a predetermined upper limit of the cycle number is reached. Testing is performed at different stress levels.

6.4.2 Loading conditions

The cyclic load shall be applied load-controlled at a frequency of $(5 \pm 0{,}5)$ Hz. The minimum value of the force shall be 5 N and the maximum force shall have the value that corresponds to the intended stress level. This value is calculated using the equation given in 6.3 where the width, b, and the thickness, h, are the mean values for the specimen to be tested. The waveform of the loading cycles shall be sinusoidal between the minimum and maximum values.

The test shall be continued until fracture occurs or the requisite number of load cycles has been applied. If large pores (diameter $\geqslant$ 1 mm) are found at the fracture surface, the test shall be repeated using an additional specimen. Repeat the tests until results are obtained from the requisite number of specimens which do not have large pores at the fracture surface. Test results shall be reported for all specimens (including those with large pores found on the fracture surface).

6.4.3 Determination of the fatigue behaviour over a wide range of stress levels (S/N curve method)

Where the performance of the cement is to be determined over a wide range of stress levels, the following procedure shall be applied.

Use five specimens per loading level and an upper limit of five million cycles. Start at a level of 80 % of the quasi static strength. The next lower level is chosen by stepping down from the 80 % level by a fixed amount (for example by 10 % of the quasi static strength). The next lower levels are chosen by stepping down by the same fixed amount. (In the example, the levels would then be 80 %, 70 %, 60 %, etc.). Continue in this way until a level is reached at which some specimens survive and others fail. In this way a minimum of four and a maximum of seven levels shall be covered.

6.4.4 Determination of the fatigue behaviour at specified stress levels (low stress method)

Where the performance is to be determined at stress levels which are thought to correspond to those expected to be encountered in clinical use the following procedure shall be applied.

Use ten specimens per stress level and an upper limit of five million cycles. Test at the stress levels, 35 MPa, 25 MPa and 20 MPa.

6.4.5 Comparison with a control material

When a comparison with a control material (usually a commercially available bone cement) is to be made, then identical test method and equipment shall be used.

6.4.6 Calculation and expression of the fatigue results (S/N curve method)

All data shall be depicted in a so-called S/N-diagram (Stress level versus Number of cycles, Figure 2, gives an example). This should be done using a semi-logarithmic scaling where the Y axis (ordinate) is subdivided linearly and represents the stresses whereas the X axis (abscissa) is subdivided logarithmically and represents the number of cycles.

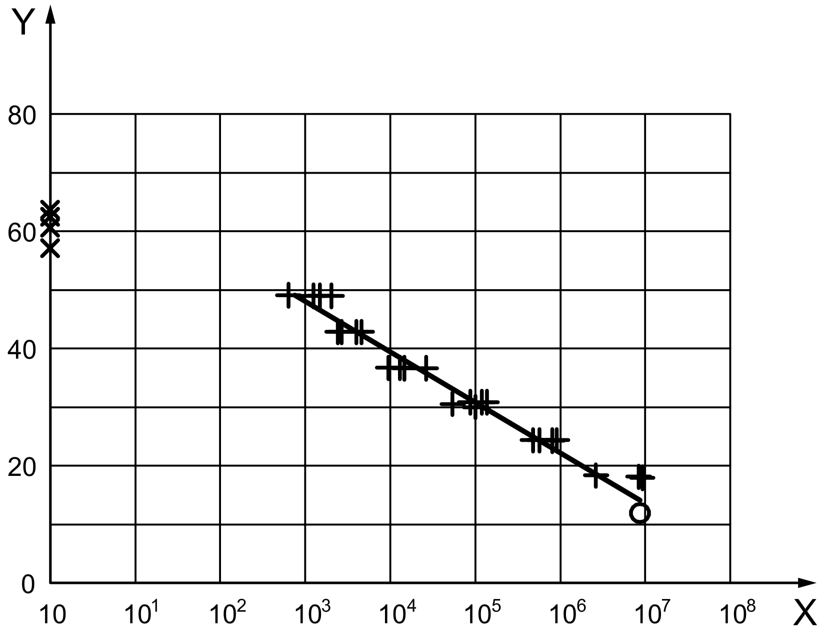
Each specimen shall be denoted in the diagram at a point which corresponds to the stress level at which it was tested, and the number of cycles which it endured either until fracture, or until the upper limit of the cycle number was reached, if the specimen survives. Using all these data points, a linear regression line shall be determined with the logarithm of the cycle number as the dependent variable. From the regression line, the mean stress at the upper limit of the cycle number is calculated. This is the “fatigue” strength for this cycle number.

For orientation purposes the quasi static data shall also be included in the diagram. In this case the points are plotted on the Y axis (which corresponds to the 1st cycle) at the appropriate stress levels. These quasi static data shall not be taken into account for the regression line.

In order to quantitatively describe the decay of the strength by fatigue, the ratio of the “fatigue” strength and the “quasi static” strength shall be calculated.

6.4.7 Calculation and expression of the fatigue results at specified stress levels (low stress method)

For the “low stress method” the median value of the life times of the specimens and the standard deviation shall be determined for each stress level.



Key

X number of cycles

Y STRESS in megapascals

× quasi static values

+ specimens failed after cyclic loading

O surviving specimen

Curve obtained by linear regression.

Figure 2 — Example for S-N-curves for a commercial bone cement

7 Test report

The test report shall include at least the following information:

a) reference to this International Standard, i.e., ISO 16402:2008;

b) identity [including batch or lot number(s), see 5.2.3] of the cement;

The following points shall be reported on both for:

i) all specimens tested and

ii) the specimens without large pores on the fracture surface;

c) the mean and the standard deviation of the values of bending strength for the “quasi static” test specimens, expressed in megapascals;

d) for 6.4.3: the S/N-Diagram;

e) for 6.4.3: the “fatigue” strength value (stress level) at five million cycles expressed in megapascals and determined from the regression line in the S/N Diagram;

f) for 6.4.3: the ratio of the “fatigue” and the “quasi static” strength, expressed as a percentage;

g) for 6.4.4: the median value and the standard deviation of the life times of each stress level;

NOTE 1 For items c) to g), the data and the S/N-curves of the evaluations for i) and ii) can be depicted partly or completely in the same diagram for purposes of comparison.

NOTE 2 If applicable, the corresponding data measured for the control bone cement can be depicted in the same diagram as the data obtained for the test material.

ICS 11.040.40

Price based on 6 pages