TECHNICAL SPECIFICATION

ISO/TS 14569-2

First edition
2001-08-15

Dental materials — Guidance on testing of wear —

Part 2:
Wear by two- and/or three body contact

Produits dentaires — Lignes directrices sur les essais de résistance à l'usure —

Partie 2: Usure par contact entre deux et/ou trois corps

Reference number
ISO/TS 14569-2:2001(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.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years with a view to deciding whether it should be confirmed for a further three years, revised to become an International Standard, or withdrawn. In the case of a confirmed ISO/PAS or ISO/TS, it is reviewed again after six years at which time it has to be either transposed into an International Standard or withdrawn.

Attention is drawn to the possibility that some of the elements of this part of ISO TS 14569 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 14569-2 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

ISO/TS 14569 consists of the following parts, under the general title *Dental materials — Guidance on testing of wear*:

— *Part 1: Wear by toothbrushing*

— *Part 2: Wear by two- and/or three body contact*

Introduction

It is well understood that the wear mechanisms in the mouth are very complex. In addition they may differ from one individual to another. Therefore it appears impossible to reproduce these varying conditions by a single wear test.

As a consequence many wear tests have been proposed in dental science. Most of them consider mainly one specific aspect of the different mechanisms, some of them even claim to be able to characterize the wear resistance of dental materials completely. However, these procedures are not really comparable because of the different wear mechanisms considered, and no generally accepted method exists.

Therefore, it makes sense to utilize laboratory tests, investigating separately the various wear aspects arising under clinical conditions. They may determine the wear only for those clinical situations in which the same wear mechanism dominates, but it might be possible to predict the complete clinical wear by a number of different test methods.

In this second part of ISO/TS 14569, the wear by occlusal contact of antagonistic teeth is considered. The intention of this part is to collect and describe the various existing laboratory tests and to define test conditions so that they can be used at least for screening different materials.

Because of the very little wear in most of the test methods, a profilometer, laser scanner or similar method is used to measure the wear. For all these tests, computer software is necessary. This software is not yet specified or standardized. It has not yet been defined to what precision the screening of the surface has to be done, nor if the whole wear pattern has to be measured or only a part from it. From a practical standpoint, the patterns must also be precisely matched before and after the test, and for this purpose reference points have to be made in some cases, especially when measuring the antagonist.

The methods collected in this part of ISO/TS 14569 thus far leave these questions open to the common sense of the person who tests, but their answers will be incorporated later when more experience exists with these test procedures.

Wear, determined according to this part of ISO/TS 14569, is only valid together with the stated combination of tested materials. A generalization of the value obtained, for example as a material constant, is not possible. Polyacrylate as reference material, as well as sintered alumina for the antagonist, does not necessarily represent the situation in the mouth. These laboratory tests only give an indication for the clinical performance.

Dental materials — Guidance on testing of wear —

Part 2:
Wear by two- and/or three body contact

1 Scope

This part of ISO/TS 14569 specifies test methods for the assessment of resistance to wear of materials occurring on the occlusal surfaces of restorations, in or on natural teeth or on artificial teeth, as a result of physiological activity in the mouth. Some of the proposed methods include wear from foodstuff as well as, or only, wear by direct contact. The test methods shown in Table 1 are described.

This part of ISO/TS 14569 is not applicable to phenomena such as the marginal degradation and loss of substance by chemical processes, swelling, splintering of edges, or wear from toothbrushing.

Table 1 — Test methods for wear included in this part of ISO/TS 14569

Clause	Test method	Antagonist	Medium	Movement	Reference	Measurement
4	DIN	Al_2O_3	H_2O	sliding	polymethyl methacrylate sheet	mass or profilometry
5	Acta	steel or dental material	rice, husks of millet spray	sliding	—	profilometry
6	Zurich	tooth enamel	H_2O	impact + sliding	last test	profilometry
7	Alabama	polyacetal	PMMA beads	impact + sliding	—	REM
8	Freiburg	Al_2O_3	H_2O	sliding	polymethyl methacrylate sheet	mass or profilometry
9	Minnesota	tooth	H_2O	sliding	—	profilometry
10	OHSU	tooth enamel	poppy seed	impact + sliding	—	profilometry + video-imaging
11	Newcastle	steatite or tooth enamel	H_2O	sliding	—	profilometry

2 Normative reference

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO/TS 14569. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO/TS 14569 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Terms and definitions

For the purposes of this part of ISO/TS 14569, the following terms and definitions apply.

NOTE See also the terms and definitions given in references [5], [7], [8] and [9].

3.1
abrasivity
ability of a material or substance to cause abrasive wear

3.2
abrasive wear
wear due to hard particles or hard protuberances forced against and moving along a solid surface

NOTE Abrasive wear can be subdivided in “two-body abrasion” and “three-body abrasion”.

3.3
two-body abrasion
abrasive wear in which the cutting asperities are fixed on one or both surfaces

3.4
three-body abrasion
abrasive wear in which the abrasive particles are loose particles in slurry

3.5
adhesive wear
wear due to localized bonding between contacting solid surfaces and leading to material transfer between the two surfaces or loss from either surface

3.6
attrition
type of two-body abrasion where teeth or restorations are in occlusal contact

NOTE In the mouth this type of abrasion is mostly the result of more than one mechanism of abrasion.

3.7
corrosive wear
wear in which chemical or electrochemical reaction with the environment is significant

NOTE Corrosive wear may result from the interaction with chemicals which have a softening effect on the surface so that the surface is rubbed away by an opposing surface (e.g. dietary erosion or from regurgitation).

3.8
erosion
〈tribology〉 progressive loss of original material from a solid surface due to mechanical interaction between that surface and a fluid, a multicomponent fluid, or impinging liquid or solid particles

3.9
fatigue wear
wear of a solid surface caused by fracture arising from material fatigue

NOTE This situation is often observed with rolling rather than sliding of surfaces.

3.10
wear
loss of material from a surface, caused by mechanical contact, movement of a solid or liquid body, chemical action or both chemical and mechanical action simultaneously

NOTE This terminology may differ somewhat from the terminology used in industry, because some mechanisms may be important in industrial processes but do not occur in the mouth. Also, wear in the mouth is usually caused by different mechanisms acting simultaneously.

4 Test method — DIN

4.1 Principle

Two specimens slide upon each other under a certain load at room temperature in water. Wear is determined by weighing the loss of substance or by other methods and reported as the worn height. Depending on the method used, the loss of substance can be very small. In this case the worn volume can be determined by scanning the surface with a profilometer, laser scanner or any other equivalent method. These guidelines for testing wear are designed for testing crown and bridge veneering resins, therefore polymethyl methacrylate (PMMA) was chosen as a reference material. This reference material is tested simultaneously and against the same antagonist material as the test material. It is always necessary to compare the value of a tested material with the value of PMMA as reference material.

4.2 Test conditions

The wear test is carried out at (23 ± 2) °C.

4.3 Apparatus and materials

4.3.1 Test equipment for wear, allowing adjustment of parameters as follows:

— pressure from the abrading antagonist against the specimen should be 8 N/mm^2 to 10 N/mm^2;

— abraded surface should be loaded and unloaded at intervals;

— speed at which the two abrading surfaces glide on each other should be not more than 100 mm/s;

— temperature of the water surrounding the specimens should be kept at (23 ± 2) °C.

4.3.2 Deionized water, in accordance with ISO 3696.

4.3.3 Antagonist material, such as densely sintered alumina or any other material, which should be tested as antagonist.

NOTE The material described in DIN/VDE 0335 Part 3 (material from Group C700 Type 799) has been found suitable.

4.3.4 Three reference specimens made from linear uncrosslinked and unplasticized PMMA with a molecular mass over 1 000 000.

NOTE Plexiglas, Perspex and Acrylite are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO/TS 14569 and does not constitute an endorsement by ISO of these products.

4.3.5 Instrument or method for determination of the worn volume of the specimens, such as an analytical balance with an accuracy of 0,1 mg.

A **profilometer** or **laser scanner** together with computer software for automatically registering data and calculating the results can also be used.

4.4 Preparation

4.4.1 Test specimens

The specimens should be prepared according to the instructions from the manufacturer. The moulds to prepare the specimens should be designed according to the requirements of the test equipment used. Six specimens should be prepared. All specimens should be ground and polished as recommended by the manufacturer. All specimens are stored at (37 ± 1) °C in water for 7 days. If wear is determined by the loss of mass, the density (ρ) of each specimen after storage in water should be determined by Archimedes' principle (see ISO 1183-1).

4.4.2 Reference specimens

Reference specimens may be cut from a linear uncrosslinked and unplasticized PMMA sheet with a molecular mass over 1 000 000.

NOTE PMMA may be used when polymer-based materials are tested, but to compare the results of ceramic or alloys also other materials may be used as reference.

4.4.3 Antagonist

If densely sintered alumina is used for the antagonist, its surface should be sandblasted with corundum [particle size (150 ± 20) µm] at 4 bar and 20 mm to 30 mm distance until the surface has a uniform and dull appearance. The mean roughness value Ra (the mean value of all distances of the roughness profile) should be approximately 0,75 µm.

4.5 Procedure

4.5.1 Gravimetric method

Remove the specimens from their storage water with tweezers, wipe with a clean dry hand-towel until free from visible moisture, wave in air for 15 s and weigh 1 min after removal from the water with a precision of 0,000 2 g. Weigh each of the six test specimens as well as the reference specimens before and after the wear test. Record the mass, in milligrams, before the wear test as m_1 and after the test as m_2.

Carry out the wear test using the equipment specified (4.3.1).

On each specimen determine the worn area with the help of a slide gauge or micrometer. Record this area as S mm^2.

4.5.2 Profilometric method

Measure the surface profile of each of the six test specimens as well as the surface of the reference specimens before and after the wear test by scanning the surface line by line. The individual sets of data recorded should be from points not more than 100 µm apart.

Carry out the wear test using the equipment specified (4.3.1).

The worn area (S) should also be recorded.

4.6 Calculation and expression of results

4.6.1 Gravimetric method

Calculate the vertical wear (H) and the relative wear (H_{ref}) as follows:

$$H \text{ in } \mu\text{m} = \frac{(m_1 - m_2) \times 1000}{\rho \times S}$$

$$H_{\text{ref}} \text{ in } \mu\text{m} = \frac{(m_1 - m_2)_{\text{ref}} \times 1000}{\rho_{\text{ref}} \times S_{\text{ref}}}$$

$$H_{\text{rel}} \% = \frac{H \times 100}{H_{\text{ref}}}$$

where

H is the height of wear from the test material, in micrometres;

H_{ref} is the height of wear from the reference material, in micrometres;

H_{rel} is the height of wear relative to the reference material.

m_1 is the mass before the wear test, in milligrams;

m_2 is the mass after the wear test, in milligrams;

ρ is the density of the test material;

ρ_{ref} is the density of the reference material;

S is the abraded area, in square millimetres, of the test material;

S_{ref} is the abraded area, in square millimetres, of the reference material.

4.6.2 Profilometric method

4.6.2.1 Worn height of the specimens

From the computerized data calculate the mean vertical wear loss, in micrometres, from the test specimens. Register the mean value (H) as the mean value of the different test specimens. Register also the maximum height measured on each specimen and calculate a mean value of all specimens as (H_{max}).

Register and calculate the mean value (H_{ref}) in the same way as above as the mean value of the different reference specimens if those were tested.

Calculate the lost height relative to the reference material as follows:

$$H_{\text{rel}} \% = \frac{H \times 100}{H_{\text{ref}}}$$

where

H is the height of wear from test material, in micrometres;

H_{ref} is the height of wear from the reference material, in micrometres;

H_{rel} is the height of wear relative to the reference material.

4.6.2.2 Worn volume

Calculate the lost volume, in cubic micrometres, from the test specimens and register the mean value (V) as the mean value of the different test specimens.

Register and calculate the mean value (V_{ref}) as the mean value of the different reference specimens which were tested.

Calculate the lost volume relative to the reference material as follows:

$$V_{rel}\ \% = \frac{V \times 100}{V_{ref}}$$

where

V is the volume of wear, in cubic micrometres;

V_{ref} is the volume of wear, in cubic micrometres, of the reference material;

V_{rel} is the volume of wear relative to the reference material.

4.6.2.3 Worn height of the antagonists

From the computerized data recorded from the antagonists, calculate the mean lost height. Register the mean value (AH) as the mean value of the different antagonists.

4.7 Test report

The test report shall at least contain the following:

a) reference to this Technical Specification, ISO/TS 14569-2;

b) the reference material (PMMA or other);

c) the material used as antagonist;

d) the mean depth of the wear pattern on the antagonist;

e) the mean depth of the wear pattern of the reference (H_{ref});

f) the mean depth of the wear pattern of the tested material (H);

g) the relative wear of the tested material (H_{rel});

h) the maximum height of wear of the tested material (H_{max});

i) the volume of wear relative to the reference material (V_{rel});

j) the density of the tested material (ρ);

k) the density of the reference material (ρ_{ref});

l) if applicable, the stylus used in the profilometry;

m) responsible person and signature.

5 Test method — ACTA

5.1 Principle

Two wheels rotate in different directions but with about 15 % difference in the circumferential speed (this is called slip) while having near contact on the circumference. Test specimens are placed on the circumference of one wheel and antagonist specimens are placed on the other wheel. Therefore several wear experiments can be performed in one run. The force with which the two wheels work against each other is adjusted to about 15 N. Both wheels are placed in a slurry of rice flour and husks of white millet spray in a buffer solution. During the wear test, which lasts about 55 h, the antagonist wheel wears a track or trough into the test specimen, leaving an untouched area at both sides as reference for the determination of the wear.

The material lost by wear can be determined by tracing each sample with a profilometer or by other equivalent methods.

The method allows determination of wear of test materials against different antagonist materials. The inclusion of a reference material in each test is optional.

5.2 Test conditions

The wear test is carried out at (23 ± 2) °C.

5.3 Apparatus and materials

5.3.1 Test equipment for wear, allowing adjustment of parameters as follows:

- pressure from the abrading antagonist wheel against the specimen wheel should be 15 N;
- speed at which the two abrading wheels rotate should be about 1 r/s and the difference in speed of the specimen surface and the antagonist surface is 15 %. The wear machine should also be equipped with a stirrer for the wear slurry and with means to prevent evaporation of water from the wear slurry.

NOTE The 15 % slip at 1 Hz to which the unit is adjusted represents about 23 mm/s, if a wheel of 48 mm diameter is used.

5.3.2 Distilled water, in accordance with ISO 3696.

5.3.3 Abrading slurry, composed of rice flour, husks of white millet spray in a buffer solution of KH_2PO_4 and NaOH and a biocide such as NaN_3 (sodium azide) or Thiomersal.

5.3.4 Profilometer or laser scanner, optionally together with computer and software for automatically registering data and calculating the results.

5.3.5 Brass sample wheel as shown in Figure 1, with an embossed or engraved mark identifying the specimen number 1, the direction of rotation, an identification of the wheel and the axis alignment.

5.3.6 Two round flanges of polytetrafluoroethylene (PTFE), having the same diameter as the wheel.

5.3.7 Set of **diamond grinding wheels** (D126, D64, D25) and a **polishing wheel** (D15).

5.3.8 Polyethylene terephthalate sheet, cyanoacrylate **glue**, SiC **sandpapers** 240-600, and black ink **marker**.