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)

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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 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to deciding whether it should be confirmed for a further three years, revised to become an International Standard, or withdrawn.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it is reviewed again after six years at which time it has to be either transposed into an International Standard or withdrawn.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.

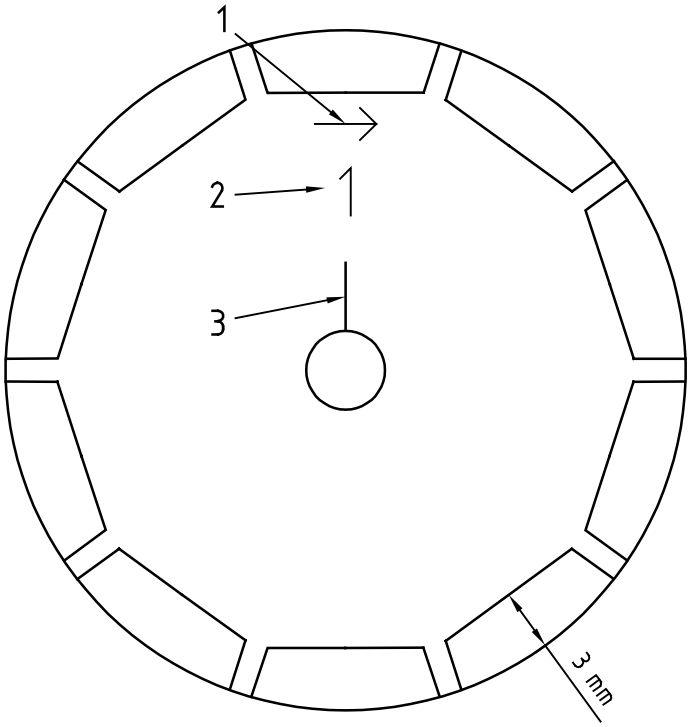
5.3.4 Profilometer or laser scanner, optionally together with computer and software for automatically registering data and calculating the results.

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**.



Key

1 Direction of rotation

2 Identification of sample 1 location

3 Axis alignment marking

NOTE This sample wheel contains 10 samples; sample 1 is located above the marked number 1. The sample width is 10 mm.

Figure 1 — Sample wheel with marking

5.4 Preparation of the test specimens

5.4.1 Preparation of the sample wheel

Place the wheel between two PTFE flanges (5.3.6) and secure it with a bolt and a wing nut.

Fill the compartments one at a time with the test material.

Cover the sample with a strip cut from the polymer sheet (5.3.8), which also covers part of the flanges so that the cylindrical shape of the wear surface on the sample is already formed.

Cure the sample following the instructions of the manufacturer.

Remove the PTFE flanges and take out the sample, unless it adheres to the wheel (some glass ionomers). Follow this procedure for all samples.

After all samples are finished, place them back with cyanoacrylate glue (3-second-glue).

Fill remaining small gaps between the samples and the wheel with glue or resin.

Wet-grind the sides of the sample wheel on 240 to 600 grit SiC sandpaper to remove excess glue and restorative flash.

Place the wheel in the wear machine with the marked side up, take care to align the markings on the axis and the wheel.

Place the roughest diamond grinding wheel (D126) in the antagonist position.

Reverse the directions of the grinding wheel by changing the switch: the LED starts blinking.

Cool the wheels by placing the glass bowl filled with water on a laboratory jack and lift until the wheels are immersed in water.

5.4.2 Grinding/polishing procedure

Start the wear machine by pushing the central reset button.

Move the wheels toward each other by turning the spacing spindle until they start touching.

When the sample wheel loses contact with the grinding wheel, move the spindle a little further. This procedure is repeated until there is contact with about 90 % of the wheel.

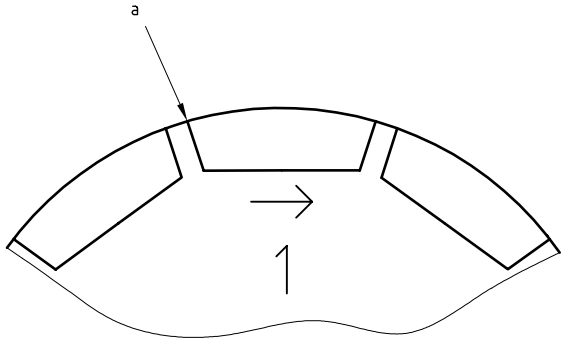
Replace the grinding wheel by wheel D64 and later by wheel D25, and continue grinding until the sample wheel is ground round completely.

Place the polishing wheel D15 and polish to generate a smooth surface.

To facilitate the grinding process, use a black waterproof marker to blacken the surface of the samples before you start grinding. This gives a good indication on the progress of the grinding process: when the samples are completely “white” again, the grinding process is finished. Repeat this before polishing with D15 to evaluate the polishing procedure.

Make a 2 mm long notch with a scalpel between sample 1 and the wall bordering to sample 10 on the reference surface (see Figure 2). This facilitates replacing the sample wheel in the profilometer set-up.

Do not rush the grinding/polishing procedure, because this will result in a facetted wheel. Take care that there is always near contact between the spacing spindle and the arm of the antagonist wheel to prevent the formation of an egg-shaped wheel.



a Make notch at arrow.

Figure 2 — Placement of the notch

5.4.3 Preparation of the antagonist wheel

Prepare the antagonist wheel in the same way as the wheel with the specimens.

5.4.4 Preparation of the slurry

Prepare a buffer solution of 41,1 g KH_2PO_4 and 9,3 g NaOH in 1 l water; add 1 g NaN_3 (sodium azide stabilizer).

Grind 120 g low-fat white rice in an electric coffee mill for 1 min. Add the flour of the rice as well as 30 g of husks of white millet spray to 275 ml of the buffer solution and mix in an electric milk shaker for 1 min while swinging the shaker to ensure complete mixing.

Transfer this mixture to the glass bowl of the test equipment.

Fill with the remaining 725 ml of buffer just before the start of the test.

5.5 Procedure

5.5.1 General

Before starting the test, control and if necessary adjust the speed, the direction of rotation of the wheels, the slip value of the wear machine and set the number of revolutions for the experiment.

5.5.2 Generating a baseline

Perform a wear-in experiment at the experimental slip conditions with 25 000 revolutions. Take care that during the wear-in experiment a complete wear track is formed. If the wear track is not complete, make an other wear-in experiment.

If it is expected that the samples will show high initial wear, reduce the wear-in experiment to 10 000 revolutions.

Trace the sample wheel and record the data as the baseline. Take care that the specimen wheel is securely fixed for the tracing and that it can be precisely repositioned after the wear test.

Mount the sample wheel in the wear machine with the markings in upward position. Take care that the radial marking on the wheel is aligned with a similar marking on the axis. Secure the wheel with a ring and a nut.

Mount the stirrer on the axis and place the plastic cover, which prevents evaporation of water from the slurry, in the correct position.

Start the wear machine and check the speed of the sample wheel by timing 30 revolutions.

Place the glass bowl containing the slurry in the correct position, set the revolutions for 200 000 and follow the instructions given for starting the wear machine.

After the test, which will take 55,5 h for 200 000 revolution at 1 Hz, lower the glass bowl with the slurry and leave it for a while so that most of the slurry can drip off the wheels and stirrer. Disassemble the stirrer and the sample wheel and clean them under running water.

Put the test specimen wheel again in the same position under the profilometer and make tracings the same way as before.

5.6 Calculation and expression of results

5.6.1 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, for the mean value of the different reference specimens tested.

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

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

where

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

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

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

5.6.2 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 lost volume from the different test specimens.

Register and calculate the mean value (V_{ref}) as the mean value of the lost volume from the different reference specimens.

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.

5.6.3 Worn height of the antagonists

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

5.7 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 (amalgam or resin composite), if one was used;

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}) if one was used;

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

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

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

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

j) the stylus used in the profilometry;

k) responsible person and signature.

6 Test method — Zürich

6.1 Principle

In a water bath with cycling temperature, the cusp of a natural tooth is pushed against the surface of a test specimen. The test specimen is fixed on a rubber socket at a 45° angle so that the antagonist glides for a short distance over the surface of the test specimen. The antagonist then moves back to start a new cycle. After about 1 200 000 cycles, a trace is formed on the test specimen, which is analysed by a scanner or profilometer or a laser scanner. The difference in the profile before and after the test is calculated using a computer program. At the end of the test, the wear of the antagonist is also investigated in the same way as for the test material.

Together with the test specimen, a reference material is tested in the same way and also against the same type of antagonist. The wear is reported as the lost height of the test specimen, as well as the worn height relative to the reference.

6.2 Test conditions

The wear test is carried out in a water bath with cycling temperatures of 2 min at 5 °C and 2 min at 50 °C.

6.3 Apparatus and materials

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

— the maximum force from the abrading antagonist against the specimen should be 49 N;

— the abraded surface should be loaded and unloaded at intervals; each cycle should follow a rounded saw curve allowing the antagonist to be in effective contact with the test specimen for 0,36 s;

— the duration of one cycle should be 0,6 s;

— the speed at which the two abrading surfaces glide on each other should be 0,56 mm/s (in this machine the speed depends on the length of the abraded trace, which itself depends on the stiffness of the rubber sockets; the horizontal excursion is usually 0,2 mm);

— the movement of the antagonist and the cycling of the water bath should be controlled and cycles counted by a computer system.

6.3.2 Two water reservoirs to supply the water bath, one kept at 5 °C and the other at 50 °C;

6.3.3 Reference specimens made from a linear uncrosslinked and unplasticized PMMA with a molecular mass over 1 000 000.

6.3.4 Six human molars with cusps of a similar shape.

6.3.5 3D-scanner and computer system, to scan and register the height of a surface area.

The scanned points on the surface should be adjustable to a distance of 50 µm. (An equivalent device measuring with the same accuracy may be used instead of the 3D-scanner.)

6.3.6 Computer and software to match the surface profile before and after the wear test and to calculate the maximum difference in height, the loss in volume, the worn surface area as well as the root mean square.

6.4 Preparation

6.4.1 Test specimens

Use the specimen holder for the preparation of the test specimens. The specimen holder has an inside diameter of 8 mm and may be filled directly with the test material and cured according to the instructions from the manufacturer. If materials are tested which cannot be cured directly in the specimen holder, use a separate mould to prepare specimens of 8 mm diameter and 2 mm height. Fix these specimens in the holder with epoxy glue or cold-curing cement.

Store the test specimens in water at 37 °C for 24 h before the test. All specimens should be wet-ground with 1 000 grade SiC paper before the test and after they have been conditioned in water at 37 °C for 24 h.

6.4.2 Antagonist

Use extracted human upper molars stored in water, with cusps of similar shape. Cut the tooth to separate the palatal cusp and fix it in the specimen holder with epoxy glue or cold-curing cement. The tip of the cusps should have a rounded, spherical shape.

If other materials are used as antagonist, prepare a specimen of a conical shape with a spherical tip. The sphere should have a radius of 1,5 mm to 2 mm and should be polished.

Store the antagonist specimens in water at 37 °C before the test.

6.5 Procedure

Mark each test specimen and reference specimen on the surface so that it can easily and accurately be repositioned for each measuring. Measure the surface profile of each test specimen as well as the surface of the reference specimens by scanning the surface line by line. The individual measurements, representing the height of the surface, are recorded as Z-values. Each measured point should be not more than 50 µm apart. A profile of a surface area of at least 4,5 mm × 4,5 mm should be measured, so that from each specimen 8 100 Z-values are recorded.

Measure the surface profile of each antagonist by scanning the surface. The individual measurements, recorded as Z-values, should be not more than 50 µm apart. A surface area of 3 mm × 3 mm should be recorded, so that from each antagonist 3 600 Z-values are recorded. If the abraded area is larger than 4,5 mm × 4,5 mm, measure the whole area.

Fix the test specimens and the antagonist samples in the machine, close the test chamber and rinse with water from the cycling system.

Divide the whole test into four steps:

— A first step with 120 000 loading cycles;

— B second step with 120 000 loading cycles;

— C third step with 400 000 loading cycles;

— D fourth step with 560 000 loading cycles.

Let the equipment run for the first step. Take the specimens, the reference specimens and the antagonists from the machine and store them in water at room temperature while waiting to be measured.

Repeat the measurement of each of the surfaces as described above. Record all data and repeat the whole test with the next cycle. Using the marks on the specimens, carefully reposition the specimens after each test for the measurement in the same way as at the beginning.

6.6 Calculation and expression of results

6.6.1 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:

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

where

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

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

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

6.6.2 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 if those 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.

6.6.3 Worn height of the antagonists

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

6.7 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 (amalgam or resin composite), if one was used;

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}) if one was used;

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

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

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

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

j) the stylus used in the profilometry;

k) responsible person and signature.

7 Test method — Alabama

7.1 Principle

In a slurry of unplasticized polymer beads in water, a stylus of polyacetal polymer is pushed against the surface of a test specimen and then rotated 30° around the long axis and back while under load. After each cycle the stylus is moved back to the original position. This cycle is repeated 1,2 times per second.

The tested material is positioned in a cavity of natural tooth.

After about 400 000 cycles, the wear pattern is tested by a replica technique under the REM as well as measures by scanning with a profilometer.

Together with the test material, a reference material is tested in the same way and also against the same type of antagonist. The wear is reported as the lost height of the test specimen as well as the worn height relative to the reference.

7.2 Test conditions

The wear test is carried out at 37 °C. One loading/unloading cycles is 0,83 s.

7.3 Apparatus and materials

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

— a flat plane stylus, machined from polyacetal with 8 mm diameter, can be moved up and down and while it is pressed against the specimen it rotates and counter-rotates through an angle of 30°;

— the maximum force from the abrading stylus (the antagonist) against the specimen should be 75 N (if the whole area of the stylus is in contact with the specimen, the load per surface area is 150 N/cm^2);

— the abraded surface should be loaded and unloaded at intervals of 0,83 s (1,2 cycles per second).

— the tangential speed of the movement of the stylus should be approximately 5 mm/s, however it depends on the surface area of the stylus which is in contact with the specimen.

7.3.2 Specimen holder, placed in a cylindrical container and kept at 37 °C.

7.3.3 Abrasive slurry, of 30 % by mass of unplasticized polymethyl methacrylate (PMMA) beads in water.

The PMMA beads should have a diameter averaging 44 µm and a molecular mass of 1 000 000.

7.3.4 Computer system, to control and count the movements of the stylus.

7.3.5 Reference specimens

Macor Ceramic in the cerammed stage is recommended.

NOTE Macor Ceramic is an example of a suitable product 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 this product. Equivalent products may be used if they can be shown to lead to the same results.

7.3.6 Six human molars, sound and caries-free, and stored in a solution of 2 % sodium azide.

7.3.7 Profilometer, to scan and register the height of a surface area.

An equivalent device measuring with the same accuracy may be used instead of the profilometer.

7.3.8 Computer and software

In the case of a large number of data points from the surface measurement, it is recommended to use a computer and software to match the surface profile before and after the wear test. With such equipment it is also possible to calculate the maximum difference in height, the loss in volume and the worn surface area as well as the root mean square of these values.

7.4 Preparation

7.4.1 Restorations

Reduce the tip of the root of the molars and mount them in the holder using a self-curing acrylic resin. Wet-grind the occlusal surface flat using a series of metallographic papers down to 600 grit. Take care that the entire surface consists of enamel.

Prepare a cylindrical cavity on the occlusal surface with a diameter of 4 mm and a depth of 3 mm. Finish all cavosurface margins with a sharp carbide bur.

Where the test material is a light-curing composite, treat the cavities according to the manufacturer's recommendations and insert the test material in three layers, cure the first two layers for 20 s and the last layer, which should be overfilled, for 60 s.

In cases where the test material is cold-curing, follow the instructions of the manufacturer for the restoration.

7.4.2 Test specimens

Where the test material is a polymerized resin, prepare a specimen of the resin with a diameter of 8 mm and directly fix it in the specimen holder, using cold-curing resin or epoxy glue.

Grind the surface of all specimens flat with wet silicon carbide paper of 600 grit and water. Take care that the occlusal surface of the specimen is horizontal and parallel to the flat end of the energy-generating stylus.

Store the test specimens in water at 37 °C for 24 h before the test.

7.4.3 Reference specimens

Prepare a cylindrical specimen of the Macor Ceramic with a diameter of 8 mm and fix it directly in the specimen holder, using cold-curing resin or epoxy glue.

7.5 Procedure

7.5.1 General

Mark each test specimen and reference specimen on the surface so that it can easily and accurately be repositioned for each measurement. Make four tracings across the surface of each test specimen as well as the surface of each reference specimen by turning them 45° after each reading, thereby producing readings every 45° around the circular specimen. If restorations are used, the actual tracings should be initiated on the enamel surface outside the contact area of the polyacetal stylus. The individual measurements, representing the height of the surface, are recorded as Z-values.

Fix the test specimens and the reference specimens in the wear machine and cover all specimens with the slurry of PMMA (7.3.3). When the specimens and the slurry have reached a temperature of 37 °C, let the machine run for 400 000 cycles (approximately 92 h).

7.5.2 Qualitative wear pattern

After the wear test, take impressions of the restorations for evaluation of the wear pattern with scanning electron microscopy. To improve the accuracy, take two impressions and discard the first one.

7.5.3 Quantitative wear

Using the marks on the specimens, carefully reposition the specimens after the test and measure as described in 7.5.1.

7.6 Calculation and expression of results

7.6.1 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 for the different reference specimens tested.

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

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

where

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

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

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

7.6.2 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 if those 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.

7.6.3 Worn height of the antagonists

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

7.7 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 (amalgam or resin composite), if one was used;

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}) if one was used;

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

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

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

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

j) the stylus used in the profilometry;

k) responsible person and signature.

8 Test method — Freiburg

8.1 Principle

The test method consists of a modified pin-on-disc test. A ceramic pin simulating the antagonistic tooth slides on a disc of the material to be tested. The material specimen is fixed in a cup-shaped holder filled with water. Both the abrading pin and the specimen in its holder rotate in opposite direction around eccentrically shifted axes. The load is applied on the pin in an axial direction.

Together with the test material, a reference material is tested in the same way.

The wear is determined by weighing the material loss, from which the wear depth is calculated by means of the material density. The wear is reported as an absolute value, as well as relative to the reference material.

8.2 Test conditions

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

8.3 Apparatus and materials

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

— the axial load is chosen such that a constant pressure of 8 MPa is produced in the contact area between pin and specimen;

— the speed at which the two abrading surfaces glide on each other should be 100 r/min (equivalent to 50 mm/s to 60 mm/s);

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

8.3.2 Deionized water, in accordance with ISO 3696.

8.3.3 Antagonistic pin, of a high-density alumina.

8.3.4 Three reference specimens, made from linear uncrosslinked and unplasticized PMMA with a molecular weight 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.

8.3.5 Analytical balance, with an accuracy of 0,1 mg, or any other instrument or method which allows the determination of the worn volume of the specimens.

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

8.4 Preparation

8.4.1 Test specimens

Five test specimens should be prepared with a diameter of 10 mm to 12 mm and a thickness of at least 2 mm. 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).

8.4.2 Reference specimens

The reference specimens should be cut from 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 other materials may also be used as reference.

8.4.3 Antagonistic pin

Cut the pin from a rod of high-density alumina with a circular cross-section of 5 mm diameter. The end surface to be in contact with the specimen shall be flat and ground such that its roughness is comparable to that of enamel.

8.5 Procedure

8.5.1 Testing

Testing is performed under the conditions given in 8.2. The maximum number of revolutions shall be 40 000. Replace the water after each 8 000 revolutions. For each material, five specimens shall be tested.

8.5.2 Weighing procedure

Before and after the test, remove the specimens from the 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 0,2 mg. Weigh each of the test specimens as well as the reference specimens before and after the test. Record the mass in milligrams before the wear test as m_1 and after the test as m_2.

On each specimen determine the worn area. Record this area as S.

8.6 Calculation and expression of results

8.6.1 Wear depth

Calculate the depth of wear H, in micrometres, and the relative wear H_{rel} 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, in micrometres, from test material;

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

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, in milligrams per cubic millimetre;

ρ_{ref} is the density of the reference material, in milligrams per cubic millimetre;

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;

8.7 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), if one was used;

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}) if one was used;

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

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

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

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

j) the stylus used in the profilometry;

k) responsible person and signature.

9 Test method — Minnesota

9.1 Principle

With the aid of a computer, an antagonist tooth moves with a masticatory, nearly sinusoidal-like movement against a lower tooth. The cycle of the movement results in a loading, sliding and unloading phase action and works with 4 Hz. The teeth (or specimens) are kept in a chamber of water at 37 °C during the test. Before and after the test the surface of the test specimen as well as the antagonist is digitized using a three-dimensional contact stylus system and computer graphics. The wear facet depths are determined by aligning the “before” and “after” digitized surfaces using a computer program developed at the University of Minnesota (USA) School of Dentistry.

The test lasts for 500 000 cycles (ca. 35 h) and, as described in the literature, tests only one specimen at a time. Specimens of shapes other than teeth can also be tested.

9.2 Test conditions

The wear test is carried out in a water bath at 37 °C. One loading cycle lasts 0,25 s.

9.3 Apparatus and materials

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

— the maximum force from the abrading antagonist against the specimen should be 13,35 N;

— the lateral excursion should be 0,62 mm;

— the cuspal contact time should be 0,23 s and the chewing rate 4 Hz;

— the profile of the load over time should be a nearly sinusoidal curve;

— a chamber around the specimens where water of 37 °C can be circulated during testing;

— a thermostatted unit for circulation of deionized water at 37 °C.

9.3.2 3D-scanner and computer system allowing a scan and registration of height of a surface area.

The software should allow the alignment of the “before” and “after” profile and calculation of the lost volume, as well as the contact area.

9.3.3 A natural antagonist or an antagonist made of a test material and in the shape of a natural cusp.

9.3.4 A reference specimen made from a linear uncrosslinked and unplasticized PMMA with a molecular mass over 1 000 000.

9.4 Preparation

9.4.1 Test specimens

Use a mould to prepare test specimens of approximately 8 mm diameter in accordance with the instructions from the manufacturer, and in a shape that can be fixed in the holder for specimens of the test machine.

The specimens can also be mounted in separate holders that fit into the test equipment.

Cut the reference specimens from a sheet of PMMA (9.3.4) and mount them in the same way as the test specimens.

Store the test specimens in water at 37 °C for 24 hours before the test. All specimens should be wet ground with 1 000 grade SiC paper before the test and after they have been in water at 37 °C for 24 h.

9.4.2 Preparation of the antagonist

Use extracted human upper molars stored in water, with cusps of similar shape. Cut the tooth to separate the palatal cusp and mount it in a specimen holder that fits into the test equipment. The tip of the cusp should have a rounded spherical shape.

The cusps can also be mounted using epoxy glue or a cold-curing cement.

If other materials are used as antagonist, prepare a specimen of conical shape with a spherical tip. The sphere should have a radius of 1,5 mm to 2 mm and should be polished.

Store the antagonist specimens in water at 37 °C before the test.

9.5 Procedure

Mark each specimen and reference specimen on the surface so that it can easily and accurately be repositioned for each measurement. Measure the surface profile of each test specimen as well as the surface of the reference specimens by scanning the surface line by line. The individual measurements, representing the height of the surface, are recorded as Z-values. The measured points should be not more than 50 μm apart. A profile of a surface area of at least 4,5 mm × 4,5 mm should be measured, so that from each specimen 8 100 Z-values are recorded.

Measure the surface profile of each antagonist by scanning the surface. The individual measurements, recorded as Z-values, should be taken not more than 50 μm apart. A surface area of 3 mm × 3 mm should be recorded, so that from each antagonist 3 600 Z-values are recorded. If the abraded area is larger than 4,5 mm × 4,5 mm, measure the whole area.

Fix the test specimens and the antagonist samples in the machine, close the test chamber and rinse with deionized water at 37 °C.

Let the equipment run for 500 000 loading cycles. Remove the specimen and the antagonist from the machine and store them in water at room temperature while waiting for the measuring procedure.

Repeat the measuring of each of the surfaces as described above. Record all data and repeat the whole test with the reference specimen in the same way. Using marks on the specimen, carefully reposition them after each test for the measurement as at the beginning.

9.6 Calculation and expression of results

9.6.1 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 height lost (wear) relative to the reference material as follows:

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

where

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

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

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

9.6.2 Worn volume

Calculate the lost volume (wear), 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 if those were tested.

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

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

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.

9.6.3 Worn height of the antagonists

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

9.7 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 (amalgam or resin composite), if one was used;

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}) if one was used;

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

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

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

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

j) the stylus used in the profilometry;

k) responsible person and signature.

10 Test method — OHSU

10.1 Principle

An enamel cusp is forced into contact with a specimen through a layer of food-like slurry. The cusp then imparts a 20 N load to the specimen and is slid across the surface over an 8 mm linear path, producing abrasive wear. At the end of the path, a static 70 N load is applied to produce localized attrition wear. This sequence is repeated at 1,0 Hz for 50 000 cycles. Each cycle consists of an active phase of 0,1 s and a resting phase of 0,9 s. The resulting wear pattern contains two distinct regions corresponding to the abrasion and attrition events. The wear patterns are analysed by a diamond-tipped profilometer making ten equally spaced passes perpendicular to the length of the wear pattern. The wear pattern consists of a zone created by abrasion wear and a zone of attrition wear. Each zone is analysed separately as well as the antagonist.

10.2 Test conditions

The wear test is carried out at room temperature. One loading cycle lasts 0,1 s.

10.3 Apparatus and materials

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

— the force from the abrading antagonist against the specimen should be 20 N while sliding over the specimen. At the end of the sliding path a load of 70 N should be applied to produce localized attrition wear;

— the speed at which the antagonist slides over the specimen should be 80 mm/s;

— the wear sequence should be repeated at 1,0 Hz for 50 000 cycles, in which each cycle includes 0,1 s of active sliding phase and 0,9 s resting phase during which the other four chambers of the equipment are active.

— a means to keep the food slurry over the specimens and prevent evaporation from the slurry.

10.3.2 Distilled water, in accordance with ISO 3696.

10.3.3 Five human molars with cusps of a similar shape.

10.3.4 Abrading slurry, composed of 3 g poppy seeds and 1,5 g PMMA beads of size range 5 μm to 125 μm in 15 ml distilled water.

To prevent spoilage, 2 mg of Thymol may be added. The poppy seeds should be ground in a mortar and pestle prior to mixing.

10.3.5 Rectangular stainless steel mould for preparation of specimens of dimensions 2,5 mm × 5 mm × 12 mm.

10.3.6 Reference specimens of dimensions 2,5 mm × 5 mm × 12 mm made from linear uncrosslinked and unplasticized PMMA with a molecular mass over 1 000 000.

10.3.7 Profilometer or laser scanner with a vertical accuracy of ± 1 μm, together with **computer and software** for automatically registering data and calculating the results.

10.3.8 Video-image analysis system to measure wear facet area on antagonist cusp.

10.4 Preparation

10.4.1 Test specimens

Use a stainless steel frame made from a plate of dimensions 2,5 mm × 25 mm × 15 mm with a central aperture of 5 mm × 12 mm. Place the frame on a polyethylene terephthalate (PET) film strip over a glass slab. Fill the aperture with the test material. Place a PET strip on top and press so that both sides of the frame are flat. Cure the test material according to the instructions of the manufacturer.

Remove the specimen from the frame, place it in the centre of an acrylic ring of 25 mm inside diameter and cast in place by filling the ring up with epoxy. When the epoxy has set, wet-grind each 25 mm disk specimen with 600 grit followed by 1 000 grit silicon carbide paper and polish with a slurry of 5 μm silicon carbide. Prepare five specimens.

Ultrasonically clean all specimens and store in water at 37 °C for 24 h before being tested.

Light-curing materials should be cured from both sides of the specimen.

10.4.2 Antagonist

Use extracted human upper molars stored in water, with cusps of similar shape. Cut the tooth to separate the palatal cusp and fix it in the specimen holder with epoxy glue or cold-curing cement. Using a diamond bur, grind the tip of the cusps into a spherical shape with a diameter of 10 mm. To exactly reproduce the 10 mm diameter sphere on the cusp, it is recommended that the handpiece with the diamond bur be mounted in a custom pivoting fixture on the toolpost of a lathe.

Finish the surface with 600 grit silicon carbide paper and then with 1 000 grit silicon carbide paper and finally polish with a slurry of 5 µm silicon carbide. If other materials are used as antagonist, prepare the antagonist specimen with the same spherical tip as for the tooth.

Ultrasonically clean the antagonist specimens and store them in water at 37 °C before the test.

10.5 Procedure

10.5.1 Mount four specimens and one reference specimen in the test chambers. Pour the slurry (10.3.4) into the chambers over the specimens. Fix the antagonist on the stylus of the wear machine and let it run for 50 000 cycles.

10.5.2 After the wear test, record the surface profile of each of the four test specimens as well as the surface profile of the reference specimen by scanning the surface, making ten equally spaced passes perpendicular to the length of the wear pattern. The individual measurements recorded should be taken at points not more than 50 µm apart. The worn area (S) of the enamel should also be computed using video-imaging analysis.

10.5.3 Compute the abrasion wear depth, HA, in micrometres, as the mean of the average depth from traces No. 4, No. 5 and No. 6. Compute the attrition wear depth, HB, as the mean of the average depth from traces No. 8 and No. 9. Perform this computation also for the reference specimen to find HA_{ref} and HB_{ref}.

10.5.4 Determine the volume lost from the abrasion region VA and VA_{ref} by performing a numeric integration of traces No. 4 to No. 6. Determine also the volume lost from the attrition region VB and VB_{ref} by performing a numeric integration of traces No. 8 and 9.

10.6 Calculation and expression of results

10.6.1 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 (HA) from the area of abrasion and the mean value (HB) from the area of attrition as the mean value of the different test specimens.

Register and calculate the mean value (HA_{ref}) from the area of abrasion and (HB_{ref}) from the area of attrition in the same way as above of the different reference specimens if those were tested.

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

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

where

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

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

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

10.6.2 Worn area on the antagonists

From the video-image analysis recorded from the antagonists, evaluate the area, in square millimetres, of the wear facet. Register the mean value (AA) as the mean value of the different antagonists worn against the test specimens, and (AA_{ref}) as the worn area on the antagonist against the reference material.

10.7 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 (amalgam or resin composite), if one was used;

c) the material used as antagonist;

d) the mean abrasion depth of the wear pattern of the tested material (HA);

e) the mean attrition depth of the wear pattern of the tested material (HB);

f) the mean area of the wear pattern on the antagonist (AA);

g) the mean abrasion depth of the wear pattern of the reference material (HA_{ref}) if one was used;

h) the mean attrition depth of the wear pattern of the reference material (HB_{ref}) if one was used;

i) the mean area of the wear pattern on the antagonist of the reference (AA_{ref}) if one was used;

j) the stylus used in the profilometry;

k) responsible person and signature.

11 Test method — Newcastle

11.1 Principle

A spherical antagonist of a ceramic material (e.g. steatite) or an enamel cusp slides under load against the surface of the test material. Wear is determined as the mean and/or maximum wear depth or the cross-sectional area of the wear scar – all determined by profilometry. Recognizing the importance of friction to the wear process, the coefficient of friction can be monitored throughout testing. Also, recognizing that the wear of the ceramic by the material is important, the wear facet area on the ceramic abrader is measured and presented.

11.2 Test conditions

Testing should be carried out at 37 °C under water (distilled ISO 3696).

11.3 Apparatus and materials

11.3.1 Test apparatus, allowing the antagonist to slide against the test material under a normal force of 15 N.

Sliding contact should be at a speed of 50 mm/min over a travel distance of 2 mm.

11.3.2 Profilometry equipment, capable of recording a profile trace across the wear scar and allowing measurement of the mean wear depth and maximum wear depths to an accuracy of 0,5 µm.

It should also be possible, from the profile, to determine the cross-sectional area of the wear profile and calculate the volume loss.

11.3.3 Travelling microscope.

11.4 Preparation

11.4.1 Test specimens

The specimens should be prepared according to the instructions from the manufacturer. The specimens should be large enough to allow testing in a manner which does not permit the antagonist to come within 1 mm of the edge of the specimen.

The mould used for making the specimen can be used as the specimen holder during testing.

NOTE A block of cast sheet PMMA having a recess 10 mm in diameter and 2 mm deep has proved useful for this purpose.

All specimens should be ground flat and polished with fine abrasives to create a surface suitable for testing and profiling. All specimens should be stored in water at 37 °C for 7 days prior to testing.

11.4.2 Preparation of the antagonist

The ceramic material or the natural tooth used as antagonist is embedded in resin for ease of mounting in the test equipment.

Sufficient ceramic material or enamel should protrude from the resin surface to allow uninhibited contact between the antagonist and the test specimen surface.

For each test, a fresh antagonist sample should be used.

NOTE A ceramic material which has been found to be suitable for use in this test is steatite. Spheres are readily available and those of 9,5 mm diameter have proved particularly useful.

11.5 Procedure

11.5.1 Wear test

An initial running-in is performed for 1 000 cycles. This is followed by a further 9 000 cycles (total 10 000) of wear testing. The surface profile is recorded at both 1 000 and 10 000 cycles by tracing across the wear track. At least five specimens for each test material should be tested.

11.5.2 Profilometry

Carry out measurements of mean and maximum wear scar depths to an accuracy of 0,5 µm. It should also be possible, from the profile, to determine the cross-sectional area of the wear profile and calculate the volume loss.

11.5.3 Antagonist wear facet

At the end of the test, the wear facet on the antagonist should be observed through a travelling microscope. Measurements of the length and width of the facets should be made to an accuracy of 10 µm. The facet area is recorded as the product of the length and width of the wear facet.

11.6 Calculation and expression of results

11.6.1 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 worn height measured on each specimen and calculate a mean value of all specimens as (H_{max}).

If a reference was included in the test, register and calculate the mean value (H_{ref}) as above as the mean value of the different reference specimens.

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

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

where

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

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

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

11.6.2 Worn area

Calculate the lost area, in square micrometres, of the wear scar from the test specimens and register the mean value (A) as the mean value of the different test specimens.

If reference specimens were tested calculate the lost area of the wear scar in the same way as for the test specimens and calculate the relative mean value (A_{ref}) as the mean value of the different reference specimens. Calculate the lost area relative to the reference material as follows:

$$A_{rel}\ \% = \frac{A \times 100}{A_{ref}}$$

where

A is the area, in square micrometres, of the wear scar;

A_{ref} is the area, in square micrometres, of the wear scar from the reference material;

A_{rel} is the area of the wear scar relative to the reference material.

11.6.3 Worn facet on the antagonists

From the computerized data recorded from the antagonists, calculate the facet area in square micrometres. Register the mean value (AA) as the mean value of the different antagonists.

NOTE Recognizing the importance of friction to the wear process, the coefficient of friction can be measured if the frictional force during testing is monitored. This can be achieved if the test specimen is mounted vertically on a load cell and the normal force during sliding is applied horizontally. The coefficient of friction can readily be measured and values at 1 000 and 10 000 cycles reported for all five specimens.

11.7 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 (amalgam or resin composite), if one was used;

c) the material used as antagonist;

d) the area of the wear facet on the antagonist (AA);

e) the mean depth of the wear pattern of the reference (H_{ref}) if one was used;

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

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

h) the lost area of the wear scar of the tested material (A), in square micrometres;

i) the lost area of wear relative to the reference material (A_{rel}) if applicable;

j) the stylus used in the profilometry;

k) responsible person and signature;

l) the frictional force measured at intervals during testing, where appropriate.

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