TECHNICAL REPORT

ISO TR 9326

First edition
1989-07-15

Implants for surgery — Partial and total hip joint prostheses — Guidance for laboratory evaluation of change of form of bearing surfaces

Implants chirurgicaux — Prothèses partielle et totale de hanche — Recommandations pour l'évaluation en laboratoire du changement de forme des surfaces articulaires

Reference number
ISO/TR 9326 : 1989 (E)

Contents

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Foreword

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

The main task of ISO technical committees is to prepare International Standards. In exceptional circumstances a technical committee may propose the publication of a technical report of one of the following types:

— type 1, when the necessary support within the technical committee cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development requiring wider exposure;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical reports are accepted for publication directly by ISO Council. Technical reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 9326, which is a technical report of type 2, was prepared by Technical Committee ISO/TC 150, *Implants for surgery*.

Annex A of this Technical Report is given for information only.

Introduction

A laboratory assessment of the "wear" of an experimental hip joint prosthesis is a critical stage in the development of a design from prototype to production. In materials science, a number of laboratory methods (such as the pin-on-disc method) are commonly used to assess the wear properties of different combinations of materials, but the geometry of the counterfaces, the loads applied, and the conditions are far removed from those that appertain to hip joint prostheses in service. In order to obtain a better representation of conditions *in vivo*, simulators have been devised (see ISO/TR 9325) in which the wear properties of prostheses can be studied.

The evaluation of the amount of wear of a prosthesis in a simulator is complicated principally by three factors:

a) the amount of wear is usually very small and difficult to quantify with precision;

b) the changes of form of the bearing surface that take place in the prosthesis during testing may be a combination of true wear and other factors such as creep and other forms of distortion;

c) the act of making wear measurements during the course of the test can itself affect the surface properties and hence the wear properties of the counterfaces.

These and other factors have to be taken into account in the preparation of standard methods of assessing the wear of hip joint prostheses under test in a hip joint simulator. In addition, the choice of method can often depend on the construction materials, the design of the prosthesis and the data that are being sought from the test. All of the currently available methods of determining wear have some disadvantages, and the reason for preparing this type 2 Technical Report is to highlight the disadvantages and advantages of a number of methods. It is hoped that this will lead to greater awareness of the problems in making measurements and offer greater comparability in the measurements themselves. It is possible that, in due time, the experience gained as a result of the use of this Technical Report will facilitate the preparation of standard methods of measuring wear.

Implants for surgery — Partial and total hip joint prostheses — Guidance for laboratory evaluation of change of form of bearing surfaces

1 Scope

This Technical Report gives guidance on the methods available for the measurement of change of form (wear, creep, plastic deformation, etc.) of the bearing surfaces of hip joint prostheses when tested in hip joint simulators.

It highlights the strengths and weaknesses of the methods in an attempt to unify experimental technique and to increase the accuracy, precision and comparability of "wear test" data.

NOTE — Atttention is drawn to ISO/TR 9325 regarding hip joint simulators.

2 Preparation of test specimens

Test and control specimens should be prepared in accordance with the specific recommendations given in the appropriate clauses of this Technical Report and in accordance with the general recommendations in ISO/TR 9325.

3 General recommendations for measuring wear

Conventional total hip joint prostheses consist of an acetabular bearing surface of polymeric or ceramic material articulating with a femoral component made of metallic or ceramic material. A change of form of the bearing surfaces will result from wear and/or creep and/or plastic deformation. Wear products may be in solution or particulate.

Wear produces a change in the dimensions of the specimen due to the removal of material with consequent loss of mass. Wear should be distinguished from other types of change of form common in polymeric materials, in which dimensional changes occur without loss of mass, e.g. creep. It is thus advisable or, in some instances depending on the method of assessment, essential to determine the amount of creep separately by a method such as subjecting a stationary component to the same loading cycle, number of loading cycles and environment as the test specimen.

It is known that visco-elastic deformation can occur. It is not considered in this Technical Report, but measurements of wear should be completed at the same time after the end of each test in order to minimize the effects of this factor.

The collection, inspection, and quantitative analysis of the particulate wear debris produced during a test is recommended, as it is often helpful in studying wear mechanisms. However, it is not recommended as the sole criterion for wear measurement.

It is often essential or desirable to examine the bearing surfaces of the specimens during the course of determining the wear, but such disassembly of the test rig and inspection of specimens should be kept to a minimum because the procedures involved are likely to influence subsequent wear mechanisms and rates.

Several established methods of estimating wear have been employed, all of which have some advantages whilst none are completely satisfactory or free from disadvantages. The most commonly used method is that of determining the loss in mass of a component, and this (and to a lesser extent other methods) are discussed in subsequent clauses.

Brief reference is also made to the evaluation of friction, since this is commonly determined by appropriate sensing and instrumentation during wear testing.

4 Measurement of wear by weighing

4.1 Preparation of specimens

In addition to the recommendations given in ISO/TR 9325, specimens that absorb water should be further prepared in order to minimize the deleterious effects of absorption on the accuracy of weighing the specimen.

Preparation of such specimens consists of soaking the specimen in lubricant (see ISO/TR 9325) at the temperature selected for the test until the mass of the component has stabilized as far as possible. Details are given in 4.2. The amount and duration of absorption varies considerably between different materials. A low-absorption material such as polytetrafluoroethylene may stabilize after 14 days soaking, whereas other materials may require 30 days and yet others may never reach an equilibrium mass.

Although soaking the test specimens before testing reduces errors due to absorption, it is strongly recommended that control specimens, generally known as "soak controls", are used. These consist of control specimens which are immersed in lubricant in the same way and for the same time as the wear test specimen but which are not subjected to wear testing, thus allowing the total change in mass of the test specimen due to absorption to be estimated.

The accuracy of the method may also be improved by the use of replicate test specimens and soak controls.

Specimens made of materials that do not absorb water from the lubricant to a significant amount may be treated differently, as detailed in 4.4.

4.2 Procedure

After fabrication and characterization, the test specimens and soak controls should be cleaned and dried, for example as described in ISO/TR 9325, and then weighed to an accuracy of $\pm$ 1 µg, a degree of sensitivity necessitated by the low wear rates of as little as 100 µg per million cycles that can be encountered. The test components and soak controls should then be immersed in lubricant (see ISO/TR 9325) for 7 days, after which the soak controls are cleaned, dried, reweighed, the increase in mass calculated, and then returned to the lubricant. This procedure should be repeated every 7 days until the masses of the soak controls have stabilized. In some instances absorption will continue for a prolonged period and it may be necessary to begin wear testing before the mass has stabilized. In such cases the value of the data from the soak controls is especially important.

The mass of the test specimen and the soak controls, as determined by weighing immediately prior to commencing wear testing, should be recorded as the initial specimen and control masses, and the progressive changes in mass of the test specimens related to these in order to determine wear.

The test specimens should be placed in the test apparatus, the lubricant added and the cyclic application of the load started. Recording of the frictional force should begin simultaneously. The test specimens should be monitored for evidence of extremely high levels or abnormal patterns of wear that could necessitate early termination of the test.

The soak controls should be placed in holders in such a manner that the total surface area exposed to the lubricant is equal to that of the test specimens when mounted in the test chamber. The soak control holders should be maintained at the same temperature [usually (37 $\pm$ 1) °C], and agitated in the same manner as the test specimen.

After the selected number of load cycles, commonly of the order of 250 000 cycles, the test specimens and soak controls should be placed in the same container, cleaned, dried and weighed. Although it is essential that all test specimens and soak controls are treated identically in order to minimize differences in fluid absorption, the effect of loading on fluid absorption by the control specimen is yet to be quantified. Further work is necessary on this topic.

The test specimens may be inspected to characterize the wear process by means such as visual, microscopic, profilometric, replication or other techniques, great care being taken to ensure that the bearing surfaces are not contaminated with any substance that could affect the subsequent wear process. Should contamination inadvertently occur, the specimen should be thoroughly cleaned before restarting the test. The test chambers should always be cleaned before refilling with fresh lubricant and replacing the test specimens. The intervals between inspections should be kept constant when carrying out comparative tests.

4.3 Calculation and reporting of results

4.3.1 Wear expressed as loss of mass

The wear of each test specimen, expressed as loss of mass δM_t, should be calculated taking into account the average change in mass of the soak controls, for instance as follows:

$$\delta M_t = (M_{t1} - M_{t2}) + (M_{s2} - M_{s1})$$

where

M_{t1} is the initial mass of test specimen;

M_{t2} is the final mass of test specimen;

M_{s1} is the average initial mass of soak controls;

M_{s2} is the average final mass of soak controls.

This procedure corrects both for systematic absorption and for random variations in the amount of surface drying at each weighing.

For metallic, ceramic, composite and some other materials, the correction required for absorption may be negligible, but this should be verified by specific investigations.

It should be noted that it is possible for debris resulting from the wear of the bearing surface of one component to become embedded in the bearing surface of the other, thus rendering the mass determination unreliable as a wear indicator.

4.3.2 Wear expressed as loss of volume

Because the density of different polymers varies considerably, it is common practice to convert mass loss to volumetric loss by dividing the corrected loss in mass of each test specimen

by the density of the polymer in order to compare wear rates. The value of density used for this calculation should be reported.

4.3.3 Wear rate

Wear rate can be expressed as

a) loss in mass per unit number of load cycles;

b) loss in volume per unit number of load cycles; or

c) depth of penetration per unit number of load cycles.

The wear rate may be calculated for each or any interval in the test and for the entire duration of the test.

If the wear rate remains approximately constant during the test, the wear rate should be calculated by the method of least squares linear regression, applied to the values of mass loss corresponding to the specific numbers of load cycles.

If the wear rate changes appreciably during the test (e.g. decrease in rate due tc the components wearing in, an increase in rate due to the onset of fatigue wear or another wear mechanism) the method of linear regression should be applied to the results from each portion of the test in order to estimate the change in wear rate.

Graphical treatment or sophisticated techniques of curve fitting and data analysis may prove helpful when analysing complex test data.

4.3.4 Wear factor

Another parameter often determined from wear data is the wear factor, k, in cubic millimetres per newton metre, derived from the equation:

$$V = Nk\int pdx$$

where

V is the volume of material removed, in cubic millimetres;

N is the number of loading cycles;

$\int pdx$ is the area under the curve obtained by plotting the values of force p, in newtons, to a base of corresponding relative movements x, in metres, in the dynamic load cycle, in newton metres.

The value of k for ultra-high molecular weight polyethylene (PE-UHMW) running against a cast cobalt-chromium-molybdenum alloy femoral head under simulator conditions is of the order of 10^{-6} $mm^3/N{\cdot}m$.

The determination of the value of k may enable direct comparisons to be made between the performance of prostheses of different designs and between data obtained with other laboratory equipment such as pin-on-plate and pin-on-disc machines.

The same data analysis techniques discussed under 4.3.3 can be applied in determining the wear factor. In addition it is necessary to report the particular form of dynamic loading curve employed.

4.3.5 Friction

If friction is measured, the average and the range of magnitude of the friction measured during the test should be calculated, either as frictional force tangential to the contact surfaces or the coefficient of friction, for each test specimen. The pattern of friction during the test, with special reference to significant changes in friction, should be tabulated and reported.

Experience has shown that friction rises temporarily after cleaning and reassembly of the test specimen and apparatus.

4.3.6 Accuracy and precision

Conclusions drawn from wear and friction tests should always be reviewed in the light of statistical treatment.

As a minimum, the individual and average values, and the 95 % confidence limits should be given. When comparing two specimens the statistical significance of any difference should be given, e.g. significantly different at the 99 % level, or not significantly different at the 95 % level.

4.4 Recommendations for materials which do not absorb water to measurable extent

Each test specimen should have a control made of the same material by the same manufacturing process, and of the same dimensions and tolerance range as the test specimen.

Before the commencement of the wearing process, both the specimen and its control should be cleaned, for example as described in ISO/TR 9325. They should then each be weighed ten times in alternation i.e. test specimen, control, test specimen, control, etc. The test specimen should then be subjected to an appropriate wearing process and the control should be stored in a desiccator. On completion of each phase of the wearing process, both the test specimen and the control should be cleaned and weighed as described above.

The loss of mass due to wear should be taken as the arithmetic mean of the ten masses subtracted from the similar arithmetic mean obtained before the commencement of the test and corrected by the apparent (if any) change in mass of the control.

5 Measurement of wear by means of dimensional changes

Because it may not be possible to determine with sufficient sensitivity the small amount of material worn from a component of a total hip joint prosthesis, other methods of assessment have been employed. Three methods are considered here, in 5.1 to 5.3.

5.1 Measurement of wear depth by displacement of components during test

In principle the wear of the acetabular cup can be monitored during the test by measuring its displacement due to wear, for

example using a linear variable displacement transducer (l.v.d.t.). In order to differentiate wear from creep or recoverable deformation, two displacement transducers must be used, one monitoring the test specimen and one monitoring a stationary control specimen that is loaded in the same way as the test specimen. The difference between the two displacement readings at any given time should then represent the wear depth. The sensitivity of this method is affected by variations in temperature, the presence of wear debris and other factors. In theory it is possible to detect wear depths of 1 micrometre, but in practice the sensitivity is likely to be one or more orders of magnitude lower.

5.2 Surface profilometry

In this method the roughnesses of the sliding surfaces are measured periodically during the test using a stylus profilometer. By reference to an unloaded and unworn part of the surface, it is possible to assess the change of form of the specimen. In particular, this technique will indicate local areas of wear rather than the overall average wear of the surfaces. However, all the precautions mentioned above concerning creep, temperature, stability, etc. still apply.

5.3 Replication

In this technique, replicas of the bearing surfaces are produced and can subsequently be studied using methods such as profilometry, microscopy, etc. However, there is the possibility that traces of the replicating medium could be left behind on the joint surfaces, which could interfere with the subsequent friction and wear behaviour of the specimens. Again, the problems concerning creep, temperature, stability, etc., may be encountered.

5.4 Summary

The advantages and disadvantages of the methods of estimating dimensional changes are presented in table 1.

6 Measurement of wear by thin layer activation

6.1 General

The technique of thin layer activation (TLA) is being used increasingly in engineering for the quantitative and continuous monitoring of wear or other forms of erosion. Although there is little experience as yet with using this technique for measuring wear in hip joint simulators, it has potential advantages over the other methods of assessment previously discussed. The technique involves the irradiation of a representative part of the component surface with a beam of energetic ions, chosen in order to activate the material by a nuclear reaction that results in the production of suitably long-lived radioactive products. It is the rate of removal of these which enables wear to be determined, either by measurement of the radioactivity of the wear debris or by the residual activity of the component. Transfer of material between surfaces in contact can also be investigated.

The nuclear reactions utilized are preferably those giving rise to an isotope that emits energetic gamma rays, since these can be counted with fewer problems due to self-absorption (for example in the fluid containing wear debris). Details of suitable reactions for the materials of widespread interest in total hip joint prostheses are listed in table 2.

6.2 Polymeric materials

Polymeric materials and components, such as polyethylene or polytetrafluoroethylene, cannot be directly activated by a beam of ions because of their sensitivity to chemical and physical degradation arising from bond-breaking. Instead, a technique of recoil activation has been developed in which a sacrificial foil target is bombarded in order that radioactive reaction products can emerge and become implanted into the component that requires activation. A convenient method is to bombard a hydrogenous foil with a 50 MeV beam of ^{7}Li ions. The kinematics of the nuclear reaction are such that the recoiling ^{7}Be nuclei are confined to a narrow cone of half-angle about 7°. Steps are taken to intercept almost all the direct beam of ^{7}Li ions by an absorber (beam stop).

The amount of radioactivity induced in components for such experiments is relatively small, being about one µCi, distributed within a controlled depth from the surface of the material. Safety precautions and regulations governing the handling of such levels of radioactivity are simple and straight-forward, although the appropriate authorization is necessary.

Nevertheless, even with the sophisticated procedure of recoil activation, the surface layers of a polymer receive irradiation to a level of approximately 2 Mrad[1)] in a typical case, and while this is within the irradiation recommended for gamma sterilization for polyethylene, it is likely to cause degradation of the

Table 1 — Summary of methods of estimating dimensional changes

Method	Displacement	Profilometry	Replication
Sensitivity	1 µm	0,5 µm	0,5 µm
Advantages	*In situ* wear data	Local wear information	Can be combined with other methods, e.g. profilometry, microscopy
Disadvantages	Temperature-sensitive	Difficult to obtain geometric wear data	Possible contamination of specimen; temperature-sensitive
Measurement technique	Measure displacement in principal load direction. Use identical transducer on control specimen.	Use fixture to hold specimen in profilometer at constant temperature.	Section replica and use profilometry, optical measurement and/or microscopy.

1) 1 Mrad = 10 000 J/kg = 10 000 Gy

more radiation-sensitive materials such as polyacetals ("Delrin"). Thin layer activation can therefore be applied only to polymers and components for which gamma sterilization to 2,5 Mrad is acceptable.

6.3 Procedure

For testing in a hip joint simulator, both the femoral component and the acetabular cup can be activated and, using a lithium-drifted germanium detector, the gamma rays from the two different activating isotopes can readily be distinguished. Wear of each component can be determined independently, and this corresponds to the true wear or removal of material rather than a change of form. The change of form is measured by profilometry. Thus the combination of measurements allows the plastic deformation or creep to be assessed independently of true wear.

In practice, if a biological serum is used to simulate body fluids, it will be necessary to replace this at regular intervals before degradation occurs. At this stage the serum can be rendered sterile by adding a measured quantity of a powerful biocide (for example sodium hypochlorite). Counting of the radioactivity, in a standard detector geometry, can be carried out later, with the appropriate corrections for radioactive decay. If the volumes of serum are large, it may be appropriate to use a re-entrant type of container (termed a Marinelli beaker) so that the liquid is held as close as possible to the gamma-ray detector.

The serum and other fluids used for washing components should be stored in sealed plastics containers after each experiment is concluded, and finally disposed of in accordance with the regulations pertaining to low-level waste. It is emphasized that, by comparison with the amounts of radioactivity used in many other branches of medicine, the amounts required for thin layer activation are very small. Gamma-emitting isotopes are the least hazardous (compared with alpha or beta emitters) and the likelihood of ingestion (via liquid or dust) is minimal in the experiments of the type described.

The likely chief advantage of thin layer activation, apart from providing a measurement of true wear rates, is its sensitivity. In a typical instance on activated titanium alloy, it is possible to measure quantitatively the removal of only 1 nm of metal, and this gives an accuracy about three orders of magnitude greater than can be achieved by usual methods of profilometry of wear scars or by gravimetry.

Table 2 — Thin layer activation of materials for hip prostheses

Material	Host element	Active isotope	Half-life days	Production route
Titanium alloy	Ti	^{48}V	16	(p, n)
		^{51}Cr	28	(α, n)
		^{46}Sc	84	(d, α)
Co-Cr-Mo alloy	Co	^{56}Co	77	(d, dn)
		^{57}Co	271	(d, d^3n)
	Cr	^{51}Cr	28	(d, dn)
Stainless steel	Fe	^{56}Co	77	(p, n)
PE-UHMW		^{7}Be	53	recoil activation by $H(^7Li, n)$