INTERNATIONAL STANDARD

ISO 18535

First edition
2016-03-15

Diamond-like carbon films — Determination of friction and wear characteristics of diamond-like carbon films by ball-on-disc method

Revêtements de carbone amorphe — Détermination des caractéristiques de frottement et d'usure des revêtements de carbone amorphe par la méthode bille sur disque

Reference number
ISO 18535:2016(E)

Contents

Page

Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*.

Introduction

This International Standard gives guidance on conducting a sliding friction and wear test in a ball-on-disc configuration to determine the friction generated and wear observed in uniaxial sliding contacts between diamond-like carbon (DLC) films and a counter body.

The tribological properties of DLC films are different from those of metallic and ceramic coatings. DLC films have the tribological characteristics of low friction and low wear against other materials. Since a DLC film is a coating, rather than a bulk material, it has a limited amount of wear and possibility of delamination. Hence, a friction and wear testing method specific to DLC films is used to determine friction and wear resistance independent of delamination. In the ball-on-disk method using a DLC-coated ball on a non-coated disk, a non-coated ball on a DLC-coated disk, or a DLC-coated ball on a DLC-coated disk, the wear of DLC is minimal compared with other materials; for this reason, it is preferable to apply the coating on the ball to measure the wear rate with a reasonable accuracy. Of course, the wear on the ball side induces a decrease in contact pressure that must be taken into account for the friction coefficient interpretation. Because of these aspects, the ball-on-disk method is ideal for conventional testing of DLC coatings.

It should be noted that there are many parameters in the sliding contact that affect the magnitude of friction and wear. The aim of performing any wear test is to simulate, as closely as possible, the conditions that occur in the real application. As the deviation between the test conditions and the application conditions becomes larger, the test results become less relevant. To add credence to the test results, the appearance of the worn surfaces from the test samples are compared with the appearance of the worn surface from the actual worn component in order to ensure that similar wear mechanisms have taken place in each case. It is intended that the recommended test conditions suggested in this International Standard be used when the application conditions are not well defined but general comparison among materials is required.

This International Standard is useful for quality control of DLC films.

Diamond-like carbon films — Determination of friction and wear characteristics of diamond-like carbon films by ball-on-disc method

1 Scope

This International Standard specifies a procedure for and provides guidance on the determination of the coefficient of friction and the specific wear rate of diamond-like carbon (DLC) films. The method specifies that the materials are tested under dry conditions in pairs in a ball-on-disc configuration.

The results of the tests are not applicable when DLC-coated parts operate in a lubricated environment.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1101, *Geometrical product specifications (GPS) — Geometrical tolerancing — Tolerances of form, orientation, location and run-out*

ISO 3274, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Nominal characteristics of contact (stylus) instruments*

ISO 3290-1, *Rolling bearings — Balls — Part 1: Steel balls*

ISO 3290-2, *Rolling bearings — Balls — Part 2: Ceramic balls*

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

ISO 80000-1:2009, *Quantities and units — Part 1: General*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
wear
progressive mass removal from the surface of solid material due to relative motion with a contacting substance or substances

3.2
wear test
method of evaluating the friction and wear performance of materials in sliding contact

3.3
ball-on-disc method
wear test method in which the sliding contact is brought about by pushing a ball specimen on to a rotating disc specimen under a constant load

3.4
friction force
resisting force tangential to the interface between two bodies, when one body moves or tends to move relative to the other under the action of a normal force pressing these bodies together

3.5
coefficient of friction
μ
dimensionless ratio of the friction force, F_f, to the applied normal force, F_p

Note 1 to entry: $\mu = F_f / F_p$.

3.6
specific wear rate
W_s
rate of material removal by wear, expressed by means of the wear volume, *V*, per unit applied normal force, F_p, and unit sliding distance, *L*

Note 1 to entry: $W_s = V/(F_p \times L)$.

4 Test materials and specimen preparation

4.1 Materials

This ball-on-disc testing method can be applied to DLC coatings. The only requirement is that the ball and disc specimens having the dimensions specified below can be prepared and that they shall withstand the stresses imposed during the testing without failure or excessive flexure.

Using the ball-on-disk method, either the ball or the disk or both the ball and disk can be used as substrates of the DLC films for testing depending on the requirements. The thickness of DLC films shall be 0,01 µm to 10 µm.

Any pertinent details of ball and disk specimen such as their dimensions, surface finish, material type, composition, microstructure, and processing treatments shall be supplied.

4.2 Ball specimen

The ball specimen shall be a true sphere of more than 5 mm in diameter or a straight rod whose end part is machined to a spherical cap. The recommended diameter of the sphere is 6 mm. The surface roughness of the specimen shall not be more than 0,1 µm *Ra* as specified in ISO 4287. The Rpk value of the surface roughness should be measured and noted in the test report. Ball sphericity shall follow ISO 3290-1 and ISO 3290-2.

4.3 Disc specimen

The disc specimen shall be more than 3 mm in thickness and be large enough to enable the testing surface to contain a sliding circle of 3 mm in diameter with a minimum of 1 mm of clearance between the circle exterior and the edge of the coated region. The surfaces of the disc shall be flat and parallel to within 0,02 mm as specified in ISO 1101. The roughness of the test surface shall not be more than 0,02 µm *Ra* as specified in ISO 4287. It is recommended that "*Ra*" be used as the symbol for roughness. The Rpk value of the surface roughness should be measured and noted in the test report. Where it is necessary to use such symbols for other parameters, these shall also be specified.

5 Apparatus

5.1 Ball-on-disc testing method apparatus shall consist of the following:

— the disc holder, for securing a disc specimen;

— the drive system, for rotating the disc;

— the ball holder, for gripping a ball specimen;

— the loading mechanism, for pushing the ball specimen on to the disc specimen;

— the equipment, for measuring the friction force and the linear wear;

— the equipment, for controlling the testing atmosphere;

— the ancillary devices for the above.

5.2 The disc holder shall rotate in a horizontal or vertical plane. The eccentricity of the rotating axis shall be less than 0,02 mm and the fluctuation at the contact point in the direction perpendicular to the disc shall be less than 0,02 mm.

5.3 The drive system shall be capable of providing a controllable sliding speed that is stable under the influence of the friction forces that are generated. The drive system shall be fitted with a revolution counter or equivalent device.

5.4 The ball holder shall firmly grip the ball specimen and have a high rigidity with respect to the stress generated at the contact point with the disc specimen.

5.5 The loading mechanism shall apply a controlled load to the ball holder directly or through a lever-arm device with attached weight or by a hydraulic or pneumatic system.

5.6 The friction force shall be measured by means of mechanisms, such as a load cell, distortion of a leaf spring or measurement of rotational torque. The measurement should not affect the frictional condition. The accuracy of friction measurement shall be ±1 % or better of the applied load. A device for measuring the linear wear is optional, but when provided, it should have a sensitivity of 2,5 µm or better.

5.7 The testing atmosphere shall be controlled to within ±2 °C of the set temperature and the relative humidity to 50 % ± 10 %. Alternatively, the testing apparatus itself can be placed in a room with conditions controlled to these limits.

5.8 If the specification of testing apparatus is different from the above, it shall be described in the test report.

5.9 Micrometre calliper shall be capable of measuring as specified in ISO 3611 or equivalent or better.

5.10 The calliper shall have a vernier scale with a vernier interval of 0,05 mm or 0,02 mm as specified in ISO 13385-1.

5.11 Micrometre microscope shall be capable of reading to the nearest 0,01 mm.

5.12 Contact stylus profilometer shall be as specified in ISO 3274 or at least equal thereto in precision.

6 Testing procedure

6.1 Treatment of specimen before test

Wash specimens ultrasonically in high purity acetone or hexane for 10 min or longer, with the testing surface downward in the case of the disc specimen. Without allowing them to dry, the specimens should be rinsed with high purity hexane and then dried for 30 min or longer in an oven set at 120 °C. Acetone and/or hexane can be replaced with other solvents or deionized water as long as clean specimen surfaces are produced at the end of the procedure. The specimens shall be stored, in the same atmosphere as that used for the wear testing apparatus, until required.

6.2 Preparation of wear test

Clamp the ball and disc specimen firmly in position in their respective holders and bring them gently into contact, then apply the set load. After the testing atmosphere has stabilized for at least 30 min, start the test by rotating the disc at the set condition.

6.3 Testing conditions for friction test

Recommended test conditions for measuring only the friction coefficient are listed as follows.

a) Applied load: 3 N.

b) Sliding speed: 0,05 m/s.

c) Sliding distance: 200 m.

d) Testing atmosphere: air at room temperature; the temperature should be controlled to within ±2 °C and the relative humidity to within ±10 % of 50 % RH.

The other test conditions listed earlier should also be followed.

6.4 Testing conditions for friction and wear test

Recommended test conditions are listed as follows, but can be changed to suit the particular needs of the measuring process. All test conditions shall be described in the test report.

a) Applied load: 5 N.

b) Sliding speed: 0,1 m/s.

The diameter of the sliding circle shall be at least 3 mm and the rotational velocity of disc holder should be determined by $\mathrm{v_r} = v/ 2\pi R$.

where

$\mathrm{v_r}$	is the rotational velocity, in rotations per second (s^{-1});
v	is the sliding speed, in metres per second;
R	is the sliding circle radius, in metres.

c) Sliding distance: 1 000 m.

d) Testing atmosphere: air at room temperature; the temperature should be controlled to within ±2 °C and the relative humidity to within ±10 % of 50 % RH.

6.5 Measurement of friction force

Measure the friction force continuously during the test and record it by using a data logger or other recording device. An appropriate system for averaging the fluctuation with rotation period shall be adopted. Before the test starts, the zero of the friction force measurement device should be checked with the specimens not in contact with one another.

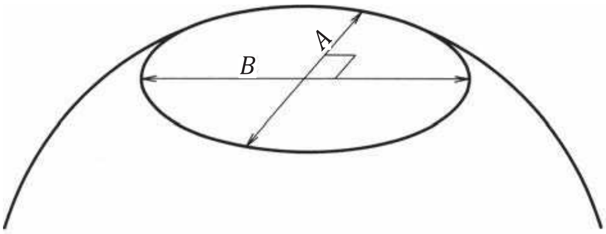
6.6 Measurement of wear scar on ball specimen

On completion of the test, there will be a roughly circular scar on the ball specimen, as shown in Figure 1. Measure the minimum diameter, *A*, and the diameter in a direction perpendicular to it, *B*, by using the micrometre microscope (5.11).

The centre of this scar can be measured using the three-point method with scar edge. If the edges of the scar are highly irregular, the figure of points shall be shown in the report.

6.7 Measurement of wear scar on disc specimen

On completion of the test, there will be a wear track on the disc specimen, as shown in Figure 2. Measure the cross-sectional profile of the wear track at four places (S1 to S4) at intervals of 90° using a contact stylus profilometer (5.12) or similar instrument and calculate the cross-sectional area of the wear track at each position.

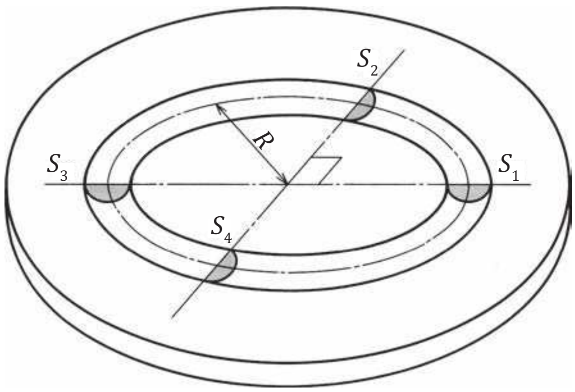


Key

A minimum diameter

B perpendicular diameter

Figure 1 — Wear scar on ball specimen



Key

R radius

S_n cross-sectional profiles where n is 1, 2, 3, or 4

Figure 2 — Wear track on disc specimen

6.8 Number of test repeats

Repeat the wear test at least three times under the same testing conditions using new specimen and balls.

7 Calculation of test results

7.1 Specific wear rate of diamond-like carbon films on ball specimen

Calculate the wear volume of diamond-like carbon film on the ball specimen using Formula (1) from the minimum diameter of wear scar and the diameter in a direction perpendicular to it, which were measured in 6.6.

$$V_{\text{ball}} = \frac{\pi A^3 B}{32D} \tag{1}$$

where

V_{ball} is the wear volume of ball specimen, in cubic metres;

A is the minimum diameter of wear scar, in metres;

B is the diameter in direction perpendicular to the minimum diameter, in metres;

D is the diameter of ball specimen, in metres.

When the wear scar on the ball is not circular due to the large depth of wear to the disc such that $B > 1{,}5\ A$, then Formula (1) shall not be applied with the wear volume obtained by 3D profilometry of the wear scar on the ball using a profilometer or similar instrument.

The specific wear rate of the ball specimen shall be obtained from V_{ball} in Formula (1) by means of Formula (2).

$$W_{\text{s(ball)}} = \frac{V_{\text{ball}}}{F_{\text{p}} L} \tag{2}$$

where

$W_{\text{s(ball)}}$ is the specific wear rate of ball specimen, in square metres per newton;

F_{p} is the applied load (normal force), in newton;

L is the sliding distance, in metres.

7.2 Specific wear rate of diamond-like carbon films on disc specimen

Calculate the wear volume of the diamond-like carbon film on the disc specimen from the cross-sectional area of wear track, which was measured as described in 6.7, by means of Formula (3).

$$V_{\text{disc}} = \frac{\pi R(S_1 + S_2 + S_3 + S_4)}{2} \tag{3}$$

where

V_{disc} is the wear volume of disc specimen, in cubic metres;

R is the radius of wear track, in metres;

S_1 to S_4 are the cross-sectional areas at four places on wear track circle in square metres.

If the ratio of the maximum to the minimum cross-sectional area exceeds 1,5, Formula (3) shall not be applied and the test is invalid.

The specific wear rate of the disc specimen shall be obtained from V_{disc} in Formula (3) by means of Formula (4).

$$W_{s(disc)} = \frac{V_{disc}}{F_p L} \quad (4)$$

where

$W_{s(disc)}$ is the specific wear rate of diamond-like carbon film on disc specimen, in square metres per newton;

F_p is the applied load, in newton;

L is the sliding distance, in metres.

In the event that the wear reaches the surface of the substrate, or the measurement result of the wear track is deeper than the thickness of the DLC film, the measurement result is not used.

7.3 Coefficient of friction

Calculate the coefficient of friction from the applied load and the average value of friction force by means of Formula (5).

$$\mu = \frac{F_f}{F_p} \quad (5)$$

where

μ is the coefficient of friction;

F_f is the average value of friction force, in newton;

F_p is the applied load, in newton.

As the coefficient of friction usually changes with sliding distance, initial, steady-state, maximum, and minimum values shall be determined and reported, when available.

In order to calculate the low-friction coefficient, the uncertainty of measurement should be calculated according to the method outlined in Annex A. In order to compare measurement results, it is important to determine the uncertainty of measurement.

When the wear reaches the surface of the substrate, the testing ball also reaches the surface of the substrate. At this time, the friction coefficient increases dramatically. When the friction coefficient increases, the test may be stopped. If the test continues after the friction coefficient increases, the data recorded after the increase in friction coefficient cannot be used in the wear resistance and friction coefficient calculation for DLC films.

Using the sudden increase in friction coefficient, it is possible to compare the wear life time of several DLC coating samples, provided all other conditions are kept identical.

7.4 Rounding off of numerical values

The specific wear rate and the coefficient of friction shall be stated to two significant digits in accordance with ISO 80000-1:2009.

8 Test report

The test report shall contain the following information:

a) a description of DLC film thickness, ball, and disk material or materials tested;

b) the specification of wear testing apparatus (disc driving method, orientation of disc rotation, loading method, friction force detecting method, atmosphere control method, etc.);

c) the testing conditions (applied load, sliding speed, diameter of sliding circle, rotational velocity of disc, sliding distance, etc.);

d) the following items which shall be reported for each individual test:

   1) the testing temperature, humidity, and their ranges;

   2) the dimensions of ball specimen and disc specimen;

   3) the substrate specimen coated with DLC film, ball, and/or disk;

   4) the wear volume of diamond-like carbon film on disc and/or ball specimen and specific wear rate calculated from this specimen;

   5) the coefficient of friction at initial, steady-state, maximum, and minimum, when available;

   6) the results of estimated uncertainty of measurement;

   7) any other matters which must be noted with regard to the state of test and the specimen after the test.

If, for the specific wear rate, a representative value under the same testing conditions is required, then the arithmetic mean of the set of wear rate values shall be used. If, for the coefficient of friction, a representative value under the same testing conditions is required, then the arithmetic mean of the set of coefficient of friction values shall be used.

Annex A
(informative)

Uncertainty of measurement for calculating of coefficient of friction

A.1 General

The friction coefficient is measured by Formula (A.1):

$$\mu = \frac{F_f}{F_p} \tag{A.1}$$

where

μ is the friction coefficient;

F_f is the friction force;

F_p is the normal force (applied on the surface).

A.2 Sources of uncertainty

A.2.1 Normal force

There are three uncertainties which can have an influence on the normal force.

— The testing weight used to apply the load on the sample surface, T_w.

— The angle deflection of the measuring arm which can induce an uncertainty on the applied load, A_d.

— The uncertainty induced by the load cell used for measuring the uncertainty related to the angle defection, L_c.

A.2.2 Friction force

There are four uncertainties which can have an influence on the friction force.

— The displacement sensor used to measure the displacement of the measuring core, correlated to the measurement of the friction force, D_s.

— The temperature fluctuations which can induce a length modification of the core inside the displacement sensor, resulting in an uncertainty on the displacement measurement, T_f.

— The calibration weight used for the calibration of the displacement sensor used to measure the friction force, C_w.

— The internal calibration procedure, including the influence of the calibration operator, which can lead to some uncertainty on the measurement of the friction force, C_p.

A.3 Uncertainty for each source

A.3.1 Normal force

— T_w: The testing weight should be calibrated and the standard uncertainty should be calculated.

— A_d: This uncertainty should be estimated by repeated measurements of the load on the sample using the load cell.

— L_c: The load cell should be calibrated and the standard uncertainty should be calculated.

A.3.2 Friction force

— D_s: The uncertainty of the displacement sensor used to measure the displacement of the measuring core should be calculated.

— T_f: The influence of the temperature on the core of the displacement sensor has been taken into account in the estimation of D_s and is therefore not taken into account.

— C_w: The calibration weight should be calibrated and the standard uncertainty should be calculated.

— C_p: From the maker specifications of the instrument, an uncertainty should be shown.

A.4 Combined standard uncertainty

A.4.1 Normal force

The combined standard uncertainty should be estimated.

$$\sum (Ui(y))^2 = (T_w)^2 + (A_d)^2 + (L_c)^2 \quad \text{(A.2)}$$

leads to

$$Uc(y) = \sqrt{\sum (Ui(y))^2} \quad \text{(A.3)}$$

A.4.2 Friction force

The combined standard uncertainty should be estimated.

$$\sum (Ui(y))^2 = (D_s)^2 + (T_w)^2 + (C_w)^2 + (C_p)^2 \quad \text{(A.4)}$$

leads to

$$Uc(y) = \sqrt{\sum (Ui(y))^2} \quad \text{(A.5)}$$

A.4.3 Friction coefficient

$$\sum (Ui(y))^2 = (T_w)^2 + (A_d)^2 + (L_c)^2 + (D_s)^2 + (T_f)^2 + (C_w)^2 + (C_p)^2 \quad \text{(A.6)}$$

leads to

$$Uc(y) = \sqrt{\sum (Ui(y))^2} \quad \text{(A.7)}$$

The combined standard uncertainty of the measured value for the friction coefficient measurement should be estimated.

A.5 Expanded uncertainty

A level of confidence of 95 % leads to the acceptance of a coverage factor of k = 2; therefore, the expanded uncertainty for the friction measurement should be calculated.

$$U(y) = Uc(y) \times \mathrm{k} \quad \text{(A.8)}$$

Bibliography

[1] ISO 13385-2, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 2: Calliper depth gauges; Design and metrological characteristics*

[2] ISO 20808, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of friction and wear characteristics of monolithic ceramics by ball-on-disc method*

[3] ISO/IEC Guide 98-1:2009, *Uncertainty of measurement — Part 1: Introduction to the expression of uncertainty in measurement*

[4] ASTM G99, *Standard test method for wear testing with a pin-on-disk apparatus*

[5] DIN 50324, *Tribology; testing of friction and wear model test for sliding friction of solids (ball-on-disc system)*

[6] JIS R1613, *Testing method for wear resistance of high performance fine ceramics by ball-on-disk method*

[7] Czichos H., Becker S., Lexow J. Multilaboratory tribotesting: Results from the Versailles Advanced Materials and Standards programme on wear test methods. *Wear*. 1987, **114** pp. 109–130

[8] Czichos H., Becker S., Lexow J. International multilaboratory sliding wear tests with fine ceramics and steel. *Wear*. 1989, **135** pp. 171–191

[9] Gee M.G., & Neale M.J. General approach and procedures for unlubricated sliding wear tests. Measurement Good Practice Guide No. 51, National Physical Laboratory, January 2002

ICS 25.220.01
Price based on 12 pages