
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Methods for evaluating wear and
friction characteristics of fine ceramic
thin films under dry and humid
conditions**

*Céramiques techniques — Méthodes pour l'évaluation des
caractéristiques d'usure et de frottement des films minces de
céramiques techniques en conditions sèches et humides*



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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.

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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Fine ceramic thin films are used in a wide variety of applications, such as sensors, actuators or other micromechanical elements; display elements; memory elements; recording media; optical elements; packaging films; and films and glass for building construction and vehicles. In the industrial application of fine ceramic thin films, resistance to wear is an important index for evaluation. The resistance to wear of fine ceramic thin films is greatly affected by environmental humidity and the humidity history of the thin film. Fine ceramic thin films are also used in different humidity environments, necessitating standards for the evaluation of wear resistance and the friction coefficient under a wide humidity range. Standards published to date concerning wear resistance testing assume only a temperature environment of 23 °C and a relative humidity of 50 %; the thickness of the thin films subject to evaluation is also comparatively large, at several to 10 µm. These testing procedures are inappropriate for evaluating the wear resistance of fine ceramic thin films that have a thickness of up to approximately 1 µm and are applied for electronic and optical devices, because the wear resistance for a smaller indentation load is affected by the relative humidity of the test environment, i.e. the mechanisms employed in the wear test for fine ceramic thin films with a smaller indentation load are strongly affected by the relative humidity of the test environment. Therefore, the wear test for fine ceramic thin films should be performed under a regulated relative humidity condition. This document provides measurement methods that facilitate the accurate evaluation of wear resistance for fine ceramic thin films in dry and high-humidity environments, where such films have a thickness of up to approximately 1 µm and are deposited on a thin substrate or an organic polymer film base. This document has been enacted to facilitate industrial development through the prompt dissemination of these measurement methods.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for evaluating wear and friction characteristics of fine ceramic thin films under dry and humid conditions

1 Scope

This document specifies a method for testing the wear resistance and friction coefficient for fine ceramic thin films in dry and high-humidity environments, where such films have a thickness of up to approximately 1 μm and are deposited on a substrate or a base, including a thin substrate or a very thin organic polymer film base.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 13565-1, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Surfaces having stratified functional properties — Part 1: Filtering and general measurement conditions*

ISO 13565-2, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Surfaces having stratified functional properties — Part 2: Height characterization using the linear material ratio curve*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

wear

phenomenon leading to progressive loss or progressive displacement from the surface of a solid material due to motion relative to a contacting body

3.2

frictional force

resistive force exerted on an opposing body when bodies in contact move or tend to slide against each other

3.3

friction coefficient

dimensionless ratio of frictional force to the normal force applied

3.4

wear test

test for evaluating friction and wear characteristics caused by sliding contact motion

Note 1 to entry: In a narrow sense, the term “wear test” implies the process of wear formation on the test specimen due to a sliding motion; in a broad sense, it implies procedures such as specimen preparation, wear formation, friction coefficient measurements, and evaluations of the wear track and the worn portion formed on the counter material.

3.5

drying chamber

hermetically sealed vessel for eliminating moisture on a test specimen surface while the test specimen remains held in a vacuum

3.6

relative humidity

ratio of water vapour partial pressure to saturated vapour pressure at a given temperature

3.7

dry air

air with a dew point of $-60\text{ }^{\circ}\text{C}$ or lower at an absolute pressure of 101,3 kPa

3.8

dry nitrogen

nitrogen with a dew point of $-60\text{ }^{\circ}\text{C}$ or lower at an absolute pressure of 101,3 kPa

4 Test specimens

Use fine ceramic thin films that have a thickness of up to approximately $1\text{ }\mu\text{m}$ and are deposited on a silicon wafer, glass, organic polymer film or other such substrate or base. Provided that the test specimens can be fitted on a wear testing apparatus as described in this document, the test specimen dimensions and shape are of no concern. However, the thickness in the area where the wear formation is performed shall be uniform.

5 Measurement principle

The wear properties of ceramic thin films are greatly affected by environmental humidity and the humidity history of the thin film. Consequently, the wear resistance of a thin film test specimen should be evaluated accurately by measuring wear of the thin film test specimen in dry and high-humidity environments.

A reciprocating wear formation method or a rotating disc wear formation method shall be used as the method for evaluating wear. Details of the principles pertaining to a reciprocating wear test method are given in EN 1071-12. Details of the principles pertaining to a rotating disc wear test method are given in ISO 20808 and EN 1071-13.

This document addresses both methods, i.e. a reciprocating wear test method and a rotating disc wear test method. [Annex A](#) provides an example of results from testing carried out using a reciprocating wear test method and a rotating disc wear test method.

6 Testing room environment

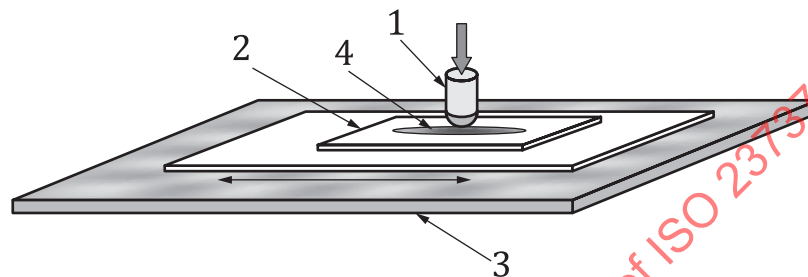
Measurements shall be carried out in a location subject to minimal changes in temperature and humidity. Specifically, testing shall be carried out under the following environmental conditions:

- a) testing room temperature: $(23 \pm 2)\text{ }^{\circ}\text{C}$;
- b) testing room relative humidity: 70 % or lower.

NOTE If the temperature of the testing room is low, condensation can form on the wear evaluation apparatus.

7 Wear formation test apparatus

7.1 Reciprocating wear tester: The reciprocating wear tester shall consist of a test specimen holder which retains a thin film test specimen, a drive apparatus which moves a thin film test specimen reciprocally, a holder which retains and secures an indenter ball, a loading mechanism which applies a constant load to the indenter ball, an equipment unit for detecting frictional force and a test environment control sealing mechanism. Figure 1 shows a schematic of a test specimen holding system for a reciprocating wear tester. Details concerning the reciprocal wear tester are given in EN 1071-12.



Key

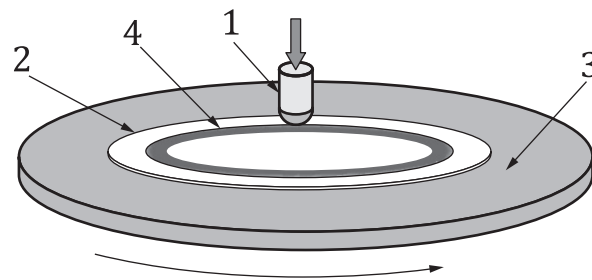
- 1 indenter with a lateral force measurement system
- 2 test specimen
- 3 test specimen holder
- 4 wear track

Figure 1 — Schematic of a reciprocating wear tester (indicating the test specimen holding system)

- a) The test specimen holder shall move reciprocally within a horizontal plane and the deflection in the direction of reciprocal motion shall be adjustable to 0,02 mm or less.
- b) The drive apparatus shall allow for the setting of a predetermined sliding speed and changes in the sliding speed due to variations in the frictional force shall be negligible. A reciprocating sliding motion counter or equivalent device shall be included.
- c) The indenter ball holder shall secure the indenter ball reliably against displacement by the frictional force generated at the area of contact between the indenter ball and the test specimen, and shall have high rigidity against induced stress.
- d) The indenter ball loading mechanism shall apply and maintain a predetermined load either directly or through a lever, and by means of a weight or a hydraulic or pressurized air system.
- e) The frictional force detection equipment can be a load cell or a leaf spring strain measurement, rotational torque measurement or other such measurement mechanism as desired, but its insertion shall not affect the frictional conditions. The measurement precision for the frictional force shall be within 1 % of the applied load. Use of an in situ linear wear measurement apparatus shall be optional. If used, the apparatus shall have a depth resolution of less than 0,01 µm.
- f) A sealing mechanism for controlling the test atmosphere shall be provided.

7.2 Rotating disc wear tester: The rotating disc wear tester shall consist of a test specimen holder which retains a thin film test specimen, a drive apparatus which moves a thin film test specimen rotationally, a holder which retains and secures an indenter ball, a loading mechanism which applies a constant load to a thin film test specimen, a frictional force detection mechanism and a test environment

control sealing mechanism. [Figure 2](#) shows a schematic of a test specimen holding system for a rotating disc wear tester. Details concerning the rotating disc wear tester are given in ISO 20808.



Key

- 1 indenter with a lateral force measurement system
- 2 test specimen
- 3 test specimen holder
- 4 wear track

Figure 2 — Schematic of a rotating disk wear tester (indicating the test specimen holding system)

- a) The rotating disc holder shall rotate within a horizontal or vertical plane, the deflection of the rotational axis shall be adjustable to 0,02 mm or less, and the deflection in the direction of the rotational axis at the area of contact shall be adjustable to 0,05 mm or less.
- b) The drive apparatus shall allow for the setting of a disc rotation speed that provides a predetermined sliding speed, and changes in the rotational speed due to variations in the frictional force shall be negligible. A rotational speed counter or equivalent device shall be included.
- c) The indenter ball holder shall secure the indenter ball reliably against rotation or displacement by the frictional force generated at the area of contact between the indenter ball and the disc test specimen, and shall have high rigidity against induced stress.
- d) The indenter ball loading mechanism shall apply and maintain a predetermined load either directly or through a lever, and by means of a weight or a hydraulic or pressurized air system.
- e) The frictional force detection mechanism can be a load cell or a leaf spring strain measurement, rotational torque measurement, or other such measurement mechanism as desired, but its insertion shall not affect the frictional conditions. The measurement precision for the frictional force shall be within 1 % of the applied load. Use of an in situ linear wear measurement apparatus shall be optional. If used, the apparatus shall have a depth resolution of less than 0,01 μm .
- f) A sealing mechanism for controlling the testing environment shall be provided.

7.3 Thermo-hygrostat chamber: The item used shall allow for the setting of relative humidity within the range of 30 % to 90 % at a temperature of 23 °C. A metal or plastic tube shall be used to introduce air of a predetermined temperature and humidity into the wear test chamber.

7.4 Dew point meter: The item shall be a capacitance (impedance) hygrometer (dew point meter) or a chilled mirror dew point meter. Details are given in JIS Z 8806.

7.5 Stylus profilometer: A stylus surface roughness measurement apparatus as specified in ISO 13565-1 and ISO 13565-2 or one with an equivalent or better precision shall be used.

7.6 Laser interferometric profilometer: The apparatus used shall make use of interference in parallel light beams with an aligned wave surface to measure the surface profile of a test specimen surface, based on the interference produced by a phase difference between light reflected from the test piece surface

and light reflected from a reference surface serving as a standard. The apparatus used shall have a height measurement resolution of approximately 10 nm at a magnification power of approximately 100× to 500×. A coherence scanning interferometry (CSI) system for three-dimensional mapping of surface height is specified in ISO 25178-604.

7.7 Optical microscope: This apparatus shall magnify an object visually for observation using optical lenses. The apparatus used shall have sufficient resolution at a magnification power of approximately 100× to 800×.

7.8 Scanning electron microscope: This microscope shall be equipped with a basic function for forming a magnified image using an appropriate method, e.g. secondary electrons obtained from a test specimen by two-dimensional scanning during irradiation of a test specimen with a tightly focused electron beam. The apparatus used shall have sufficient resolution at an observational magnification power of the order of 5 000×. For ceramic thin films, applying an electroconductive coating prior to the observation is strongly recommended.

7.9 Energy dispersive X-ray spectroscopy: This instrument shall be equipped with an energy dispersive X-ray (EDX) spectrometer to provide elemental identification by measuring the energy of X-rays emitted from a specimen due to excitation by the primary electron beam. An elemental distribution map can be acquired by combining the EDX signal with the position signal of the scanning primary electron beam.

7.10 Drying chamber: Provided that the chamber can maintain an internal pressure of 4 kPa or lower and a temperature of 130 °C or higher, its material, form and other details are not of concern.

7.11 Exhauster: A diaphragm pump or similar item shall be used. Provided that the item has an ultimate pressure of 4 kPa or lower, its type shall not be of concern.

8 Test specimen preparation

Use the same lot or the same batch to prepare thin film test specimens as needed, allowing for completion of at least three test runs.

9 Test specimen pretreatment

9.1 General

Pretreat test specimens as needed. If test specimens have been exposed to high humidity, for example cleaned using water or subject to similar processes, the test specimen shall be dried.

9.2 Test specimen cleaning

If thin film test specimens are dirty, clean them using an organic solvent or a surfactant. If an organic solvent is used for cleaning, rinse with a sufficient amount of a clean organic solvent and dry naturally or steam dry with an organic solvent. If a surfactant is used, rinse with a sufficient amount of water and dry rotationally or by, for example, dry air or hot air drying to remove water. If water is used in the cleaning, the drying treatment described hereafter shall be performed. If a mineral spirit is used in cleaning, the solvent shall be only a single boiling point spirit.

9.3 Test specimen drying

Place a thin film test specimen in the drying chamber, bring the pressure in the drying chamber to 4 kPa or lower and the test specimen temperature to 130 °C or higher, and maintain these conditions for 30 minutes. If the substrate cannot be heated to 130 °C or higher, bring the test specimen temperature to the highest temperature to which the substrate can be heated. After the drying procedure has been

completed, transfer the thin film test specimen promptly to a test apparatus for wear evaluation testing in a dry environment.

10 Environmental conditions for the formation of the wear and the evaluation of friction coefficient

10.1 General

The environmental conditions for the formation of the wear and the evaluation of friction coefficient shall be as follows:

10.2 Dry environment

Replace the atmosphere in the wear test chamber with dry nitrogen or dry air to create a dry environment under conditions where the temperature is $(23 \pm 2) ^\circ\text{C}$ and the relative humidity is 10 % or lower. Calculate the relative humidity from the dew point (frost point) measurement results.

10.3 High-humidity environment

Create a high-humidity environment in the wear test chamber under conditions where the temperature is $(23 \pm 2) ^\circ\text{C}$ and the relative humidity is 70 % to 80 %. To create a high-humidity environment, introduce air with a predetermined temperature and humidity from the thermo-hygrostat chamber into the test specimen holding area of the wear tester. Measurements can be performed using the entire measurement room as a high-humidity environment.

11 Wear motion conditions

11.1 Reciprocating wear conditions

- a) **Wear area:** This area shall be the centre of the thin film test specimen.
- b) **Stability of test specimen securing area:** The test specimen holder shall rotate within a horizontal or vertical plane, the deflection of the rotational axis shall be adjustable to 0,02 mm or less and the deflection in the direction of the rotational axis at the area of contact shall be adjustable to 0,05 mm or less.
- c) **Test specimen securing method:** Test specimens shall be pressed from above to prevent any rattling movement. If the substrate or base is thin, use a vacuum chuck to provide uniform suction retention. In the case of retention by suction, due consideration shall be given to the flatness of the test specimen surface after retaining by suction, as indicated in [Annex B](#).
- d) **Indenter ball material, diameter and surface roughness:** The indenter ball material shall be 304 stainless steel (ISO 4301-304-00-I, EN 10088 X5CrNi18-10 or JIS SUS304), 440C stainless steel (ISO 4023-440-04-I, EN 10088 X105CrMo17 or JIS SUS440C), Al_2O_3 (purity: > 99,99 %; hardness: > 1 500 HV), SiC (purity: > 99,9 %; hardness: > 2 200 HV), Si_3N_4 (ISO 3290-2; purity: > 95,0 %; hardness: > 1 600 HV) or another such material. The ball specimen shall be a true sphere with a diameter of 5 mm to 20 mm. The recommended ball diameter is 10 mm. The surface roughness of the indenter ball should preferably be 0,01 μm or less. The surface roughness (R_a – arithmetical mean roughness value) of the indenter ball shall be determined as needed by measuring the roughness curve at the equator of the indenter ball using a measuring instrument as specified in ISO 3274 and determining the mean roughness on the centre line. Pay due attention to the fact that the test results can differ depending upon the combination of the thin film test specimen material and the indenter ball material that is used. [Annex C](#) provides examples of wear testing results produced by different combinations of thin film test specimen materials and indenter ball materials.

- e) **Indenter ball cleaning:** Clean the indenter ball in pure water prior to use by performing ultrasonic cleaning, and dry with warm alcohol or alcohol steam. If a mineral spirit is used in cleaning, the solvent shall be only a single boiling point spirit.
- f) **Indenter ball load:** The indenter ball load shall be approximately 0,50 N or lower. An appropriate indenter ball load for the fine ceramic thin film test specimen should be determined by pre-testing. [Annex D](#) presents results from reciprocating wear testing with varying loads.
- g) **Reciprocating movement frequency:** 1 Hz.
- h) **Indenter ball movement speed:** 33,3 mm/s.
- i) **Indenter ball movement stroke:** 10 mm.
- j) **Wear oscillating cycles:** 1 000 oscillating cycle. In the case of low wear, the cycle number can be increased to 5 000 cycles. In the case of high wear, where the coating thickness is worn down to the substrate, 500 cycles are sufficient.
- k) **Number of tests:** Test at least three specimens under identical conditions.

11.2 Rotating disc wear conditions

- a) **Test specimen wear area:** This area shall be the centre of the thin film test specimen.
- b) **Stability of test specimen securing area:** The test specimen holder shall rotate within a horizontal or vertical plane, the deflection of the rotational axis shall be adjustable to 0,02 mm or less, and the deflection in the direction of the rotational axis at the area of contact shall be adjustable to 0,05 mm or less.
- c) **Test specimen securing method:** Disc-shaped testing specimens shall be secured in a disc holder. If the substrate or base is thin, use a vacuum chuck to provide uniform suction retention. In the case of retention by suction, due consideration shall be given to the flatness of the test specimen surface after retaining by suction, as indicated in [Annex B](#).
- d) **Indenter ball material, diameter and surface roughness:** The indenter ball material shall be 304 stainless steel (ISO 4301-304-00-I, EN 10088 X5CrNi18-10 or JIS SUS304), 440C stainless steel (ISO 4023-440-04-I, EN 10088 X105CrMo17 or JIS SUS440C), Al_2O_3 (purity: > 99,99 %; hardness: > 1 500 HV), SiC (purity: > 99,9 %; hardness: > 2 200 HV), Si_3N_4 (ISO 3290-2; purity: > 95,0 %; hardness: > 1 600 HV) or another such material. The ball specimen shall be a true sphere with a diameter of 5 mm to 20 mm. The recommended ball diameter is 10 mm. The surface roughness of the indenter ball should preferably be 0,01 μm or less. The surface roughness of the indenter ball shall be determined as needed by measuring the roughness curve at the equator of the indenter ball using a measuring instrument as specified in ISO 3274 and determining the mean roughness on the centre line. Pay due attention to the fact that the test results can differ depending upon the combination of the thin film test specimen material and the indenter ball material that is used. [Annex C](#) provides examples of wear testing results produced by different combinations of thin film test specimen materials and indenter ball materials.
- e) **Indenter ball cleaning:** Clean the indenter ball in pure water prior to use by performing ultrasonic cleaning, and dry with warm alcohol or alcohol steam. If a mineral spirit is used in cleaning, the solvent shall be only a single boiling point spirit.
- f) **Indenter ball load:** The indenter ball load shall be approximately 0,50 N or lower. An appropriate indenter ball load for the fine ceramic thin film test specimen shall be determined by pre-testing. [Annex D](#) presents results from reciprocating wear formation with varying loads.
- g) **Sliding radius:** More than 12,5 mm (if less, the curvature of the wear track can affect results).
- h) **Indenter ball sliding speed:** 33 mm/s.

- i) **The number of sliding revolutions:** 2 000 rotational cycles (revolutions). In the case of low wear, the cycle number can be increased to 10 000 cycles. In the case of high wear, where the coating thickness is worn down to the substrate, 1 000 cycles are sufficient.
- j) **Number of tests:** Test at least three test specimens under identical conditions.

12 Evaluation of wear track cross-section formed on thin film test specimen surface

Perform ultrasonic cleaning with pure water before performing an evaluation of the wear track cross-section. The cleaning time shall be 10 minutes. In ultrasonic cleaning, oscillate the test specimen to provide for uniform cleaning. After ultrasonic cleaning has been completed, dry the test specimen. The drying shall be performed by immersion in a warm alcohol bath or exposure to alcohol steam followed by natural drying, rotational drying or other such means.

Profile the cross-section of the wear track formed on the thin film test specimen by using a stylus profilometer or a laser interferometric profilometer, as specified in [Clause 7](#). [Annex E](#) provides an example of the method for quantifying a wear cross-section.

Measurements shall be performed at three points, generally near 3 mm, 5 mm and 7 mm, in reciprocating wear formation and at four points with an interval of 90° within the wear circle in rotating disc wear formation. If the formation of extreme excrescences or similar features is observed, change the measurement points or perform the ultrasonic cleaning again to obtain a grooved wear mark profile.

Calculate the wear volume from the cross-sectional area for rotating disc wear formation by using [Formula \(1\)](#), if needed.

$$V = \frac{\pi \times R (S_1 + S_2 + S_3 + S_4)}{2} \quad (1)$$

where

V is the wear volume of thin film test specimen (m³);

R is the radius of the wear track (m);

S_1 to S_4 represent the cross-sectional area at four places on the wear track circle (m²).

13 Evaluation of volume of worn portion on indenter ball surface

13.1 General

Observe the morphology of the worn portion on the indenter ball surface. From the results of the morphology observation, the volume of the worn portion shall be evaluated by the following methods.

13.2 Observation of indenter ball wear surface profile

Observe the worn portion on the indenter ball surface by using a laser interferometric profilometer, an optical microscope or a scanning electron microscope, as specified in [Clause 7](#).

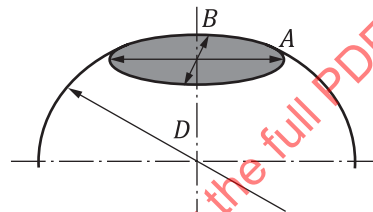
13.3 Calculation of volume of worn portion on indenter ball surface

Calculate the volume of the worn portion on the indenter ball surface by using [Formula \(2\)](#), based on the shortest diameter of the worn portion mark measured in accordance with [14.2](#) and a diameter perpendicular to the same. [Figure 3](#) shows the schematic of the worn portion on the indenter ball.

$$V_{\text{ball}} = \frac{\pi A^3 B}{32D} \quad (2)$$

where

- V_{ball} is the indenter ball wear volume (m³);
- A is the shortest diameter of the worn portion (m);
- B is the diameter in the direction perpendicular to the shortest diameter of the worn portion (m);
- D is the indenter ball diameter (m).



Key

- A shortest diameter of the worn portion (m)
- B diameter in the direction perpendicular to the shortest diameter of the worn portion (m)
- D indenter ball diameter (m)

Figure 3 — Quantification of the wear volume of the indenter ball

This formula is an approximation which simplifies the complex three-dimensional profile of a wear mark. If the wear mark profile is greatly distorted ($B > 1,5 A$), for reasons such as a deep wear mark in the sliding area of a thin film test specimen, this formula shall be deemed inapplicable. Refer to EN 1071-13.

14 Investigations of morphology and elemental distribution maps for wear tracks on thin film test specimens and worn sections on indenter balls

14.1 General

Investigate the morphology and elemental distribution maps for the wear tracks formed on the thin film test specimen surface and the worn sections formed on the indenter ball surface. From the results of the morphology and elemental distribution map investigations, the debris adhesion on the wear tracks and the worn sections on the indenter ball shall be evaluated.

14.2 Observation of the morphology

Observe the morphology of the wear track surface and the indenter ball surface by using a scanning electron microscope, as specified in [Clause 7](#). The observational magnification shall depend on the

wear track width, but it shall generally range from 500× to 2 000×. [Annex F](#) provides examples of observations of the morphology of the wear track surface and the indenter ball surface.

14.3 Investigations of elemental distribution maps

Investigate elemental distributions on the wear track surface and the indenter ball surface by using energy dispersive X-ray spectroscopy, as specified in [Clause 7](#). [Annex F](#) provides examples of elemental distribution maps of the wear track surface and the indenter ball surface.

15 Calculation of friction coefficient

In the wear formation process, the friction coefficient can be obtained as an extraneous value. Calculate the friction coefficient by using [Formula \(3\)](#) based on the applied load and the mean frictional force.

$$\mu = \frac{F_f}{F_n} \quad (3)$$

where

μ is the friction coefficient;

F_f is the mean frictional force (N);

F_n is the applied load (N).

In reciprocating wear motion, the dynamic friction coefficient and static friction produced by approximately 10 sliding strokes shall be measured as the friction coefficients for the starting wear, interim wear and ending wear, and their mean shall be taken (e.g. 1 000 measurements of reciprocating wear are taken as strokes 0 to 10, 501 to 510 and 901 to 910).

In measurements for rotating disc wear motion, the friction coefficient shall be obtained by measuring it continuously. Alternatively, the dynamic friction coefficient shall be calculated for the starting wear, interim wear and ending wear rotations, and their mean shall be obtained (e.g. 2 000 measurements of rotating wear are taken as rotation laps 0 to 10, 1 001 to 1 010 and 1 801 to 1 810).

16 Testing procedures

16.1 Measurement of thickness of thin film test specimen

Measure the thickness of each thin film test specimen and the uniformity of its thickness. A method for measuring this thickness is not designated in this document, but a method with adequate precision shall be used. For an example, refer to ISO 4518.

16.2 Thin film test specimen preparation and cleaning

Prepare the thin film test specimen and pretreat it by using the methods set forth in [Clauses 8](#) and [9](#).

16.3 Setting of wear formation conditions

16.3.1 General

Set the conditions for the wear formation.

16.3.2 Wear formation conditions for a dry environment

Place the test specimen in a wear test chamber and replace the atmosphere in the wear test chamber with dry air to bring the atmospheric temperature to $(23 \pm 2)^\circ\text{C}$ and the relative humidity to 10 % or lower. Maintain the test specimens in the aforementioned environmental conditions for 20 minutes before starting the motion for wear formation. Dry nitrogen can be used to create these environmental conditions. In that case, it has to be reported as specified in [Clause 18](#).

16.3.3 Wear formation conditions for a high-humidity environment

Place the test specimen in a wear test chamber and then control the atmosphere in the wear test chamber to bring the temperature to $(23 \pm 2)^\circ\text{C}$ and the relative humidity to 70 % to 80 %. Maintain the test specimen in the aforementioned environmental conditions for 20 minutes before starting the motion for wear formation.

16.4 Wear formation

16.4.1 Start of wear formation on the thin film test specimen

After bringing the indenter ball into contact with the test specimen and applying the test load to the ball, start the rotating or sliding motion for wear formation. Ensure that the temperature and relative humidity inside the wear test chamber are maintained at the selected conditions.

16.4.2 Friction force measurement

Where appropriate, measure the friction force during the wear formation and record it.

16.5 Evaluation of thin film test specimen surface and indenter ball surface after wear formation

16.5.1 General

Evaluate the thin film test specimen surface and the indenter ball surface after the wear motion has been completed.

16.5.2 Measurement of wear track cross-section on thin film test specimen

Measure the wear track cross-section on the thin film test specimen by using the method set forth in [Clause 12](#).

16.5.3 Evaluation of volume of worn portion on indenter ball surface

Evaluate the volume of the worn portion on the indenter ball surface by using the method set forth in [Clause 13](#).

16.5.4 Investigations of morphology and elemental distribution maps

Investigate morphology and elemental distribution maps for the wear track on the thin film test specimen and for the worn section of the indenter ball by using the method set forth in [Clause 14](#).

16.6 Calculation of friction coefficient

Calculate the friction coefficient by using the method set forth in [Clause 15](#).

17 Precautions for summarization of test results

In this testing, any non-uniformity among the thin film test specimens or inconsistencies in the evaluation conditions can cause discrepancies in the data. If necessary, increase the number of strokes or test specimens, and if the measurement data are judged to include abnormal values, complete an outlier test and discard the data. The recommended outlier test method is a Grubbs' test in accordance with ISO 5725-1 and ISO 5725-2.

18 Test report

The following items should be reported in the test results:

- a) a reference to this document (i.e. ISO 23737:2021);
- b) test method and test apparatus (test specimen movement method, loading method and friction force detection method);
- c) date of measurement, measurement operator and temperature or relative humidity of testing room;
- d) material, thickness and initial surface roughness of thin film test specimen;
- e) material of substrate or base;
- f) model and specifications of wear tester;
- g) indenter ball material and diameter;
- h) holding method for substrate or base;
- i) model and specifications of surface profile evaluation apparatus;
- j) test conditions [vacuum drying conditions and time, test environment dew point (frost point) and relative humidity and humidity level in high-humidity environment];
- k) number of test specimens;
- l) number of oscillating cycles or rotating evolutions;
- m) wear track cross-section of thin film test specimen or wear volume of thin film test specimen;
- n) friction coefficient;
- o) volume of worn portion formed on indenter ball surface;
- p) images observed for wear track on thin film test specimen and elemental distribution images if possible;
- q) images observed for worn portion on indenter ball surface and elemental distribution images if possible;
- r) other notable details with regard to evaluation status.

Annex A (informative)

Example of reciprocating slide wear results and rotating slide wear results

A.1 Difference between reciprocating slide wear results and rotating slide wear results

In this document, the methods used for both reciprocating slide wear motion and rotating slide wear motion are standardized. In these motions, the wear behaviour is different because the adhesion of debris to the test specimen or to the indenter ball, which strongly affects the wear behaviour, depends on the sliding mode. However, the fundamental data obtained to construct this document indicate the same tendency in both reciprocating slide wear motion and rotating slide wear motion under dry and humid conditions.

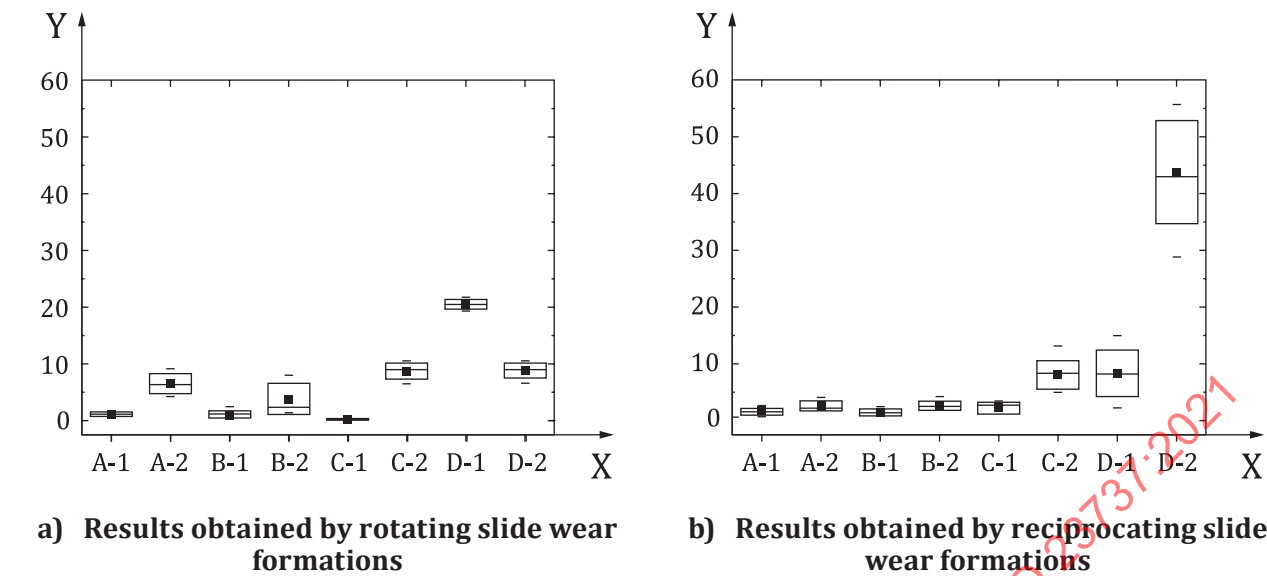
A.2 Example of reciprocating slide wear results and rotating slide wear results

[Figure A.1](#) shows a comparison of the results for a reciprocating slide wear motion and a rotating slide wear motion for as-deposited indium tin oxide thin films (as-deposited ITO: ITO-A), 300 °C-annealed indium tin oxide thin films (annealed ITO: ITO-B), Si_3N_4 thin films and SiO_2 thin films under dry conditions (relative humidity: 10 %) and humid conditions (relative humidity: 80 %). The details of thin film test specimens are shown in [Table A.1](#). The indenter ball material was 440C stainless steel. Both wear formations were performed by using a wear test instrument in which any of the rotational and reciprocating modes for sliding wear formation can be selected. Although the behaviour of SiO_2 thin film test specimens under dry and humid conditions was inversed in the wear cross-section compared to other thin film test specimens, the other test specimens exhibited the same tendency, i.e. the wear cross-section was larger for the humid condition tests. It is concluded that the tendency identified in wear tests for both reciprocating sliding and rotating sliding is unaffected by any difference in the sliding mode for rotating and reciprocating.

Table A.1 — Thin film test specimens used in sliding wear tests

Thin film test specimens	Thickness nm	Substrate	Deposition method	Deposition temperature	Post deposition anneal
As-deposited indium tin oxide (ITO-A)	300	Non-alkali glass	Sputtering	RT ^a	None
300 °C-annealed indium tin oxide (ITO-B)	300	Non-alkali glass	Sputtering	RT ^a	300 °C atmosphere
Si_3N_4	300	Non-alkali glass	Reactive sputtering	RT ^a	None
SiO_2	300	Non-alkali glass	Reactive sputtering	RT ^a	None

^a RT represents room temperature.



Y wear track cross-section (µm²)

- average
- max-min
- standard deviation

- A-1 10 % RH-ITO-A
- A-2 80 % RH-ITO-A
- B-1 10 % RH-ITO-B
- B-2 80 % RH-ITO-B
- C-1 10 % RH-Si₃N₄
- C-2 80 % RH-Si₃N₄
- D-1 10 % RH-SiO₂
- D-2 80 % RH-SiO₂

NOTE The indenter ball material was 440C stainless steel. RH represents relative humidity.

Figure A.1 — Wear cross-section obtained for several coating materials

Annex B

(informative)

Method of holding polymer film or thin glass plate with a thickness of $< 200 \mu\text{m}$ in sliding wear formations

B.1 Necessity to consider a method of holding film or thin glass plate substrates with a thickness of $< 200 \mu\text{m}$ in sliding wear formations

In sliding wear, it is necessary to hold a substrate with sufficient flatness to obtain reproducible test results. From this viewpoint, an examination shall be performed prior to the sliding test to determine whether a thin film or a thin glass plate substrate can be held with sufficient flatness to obtain reproducibility in the test. In this annex, a method involving the use of a vacuum plate chuck to hold a thin polymer film or a glass plate substrate will be considered.

B.2 Example of a holding method using a vacuum chuck plate

[Figure B.1](#) shows an image of a vacuum chuck with a diameter of 150 mm. The vacuum chuck shown in [Figure B.1](#) has a pore size of $0,5 \mu\text{m}$ and a porosity of 25 %. The surface of the chuck was polished using a #2000 diamond grinding wheel.

The surface vacuum chuck shall be cleaned by a combination of ultrasonic cleaning and backward blow prior to the chucking of a substrate. Particulates need to be removed to avoid the formation of an unevenly distributed convex curvature on the substrate surface after vacuum chucking.

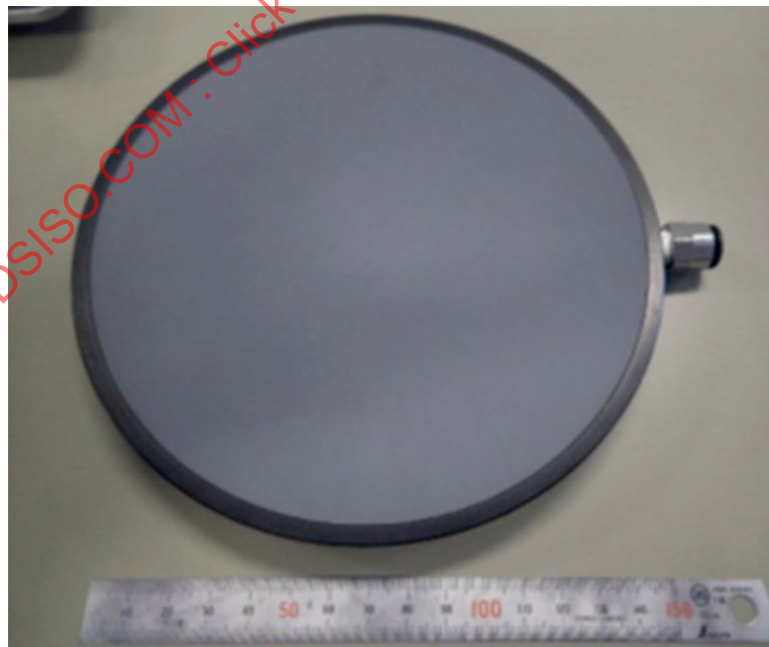
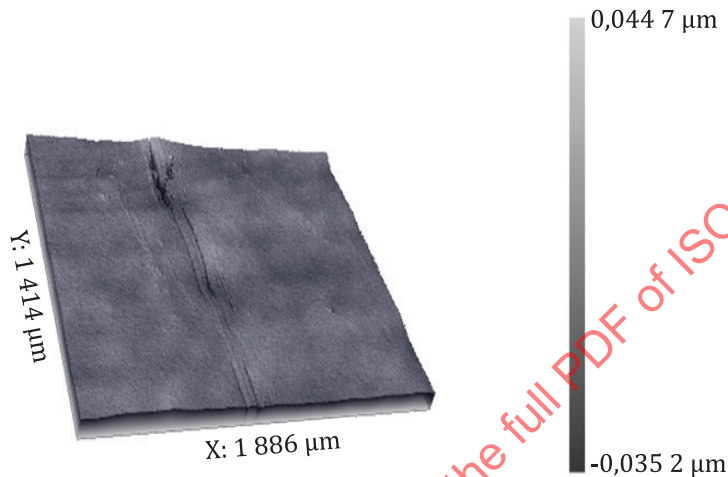


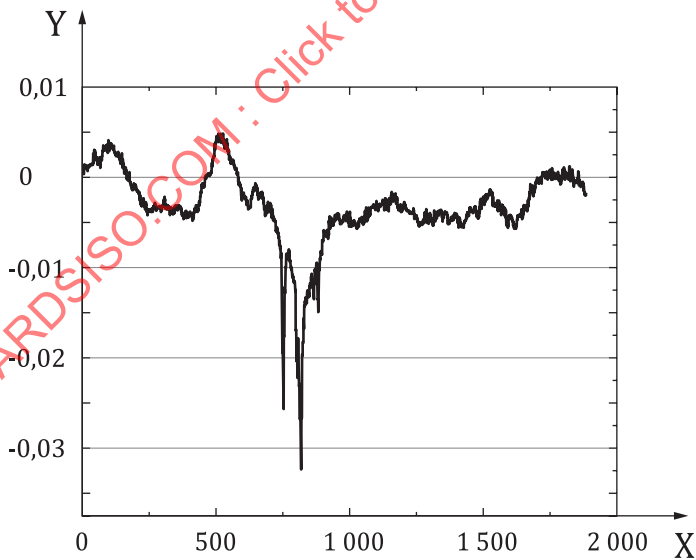
Figure B.1 — Image of a vacuum chuck used to hold a polymer or thin glass substrate with a thickness of $< 200 \mu\text{m}$

B.3 Results of a reciprocating sliding wear formation for an ITO thin film deposited on a PET film under humid conditions

Figure B.2 a) shows a surface image of an ITO-coated PET film after a reciprocating sliding wear formation was performed under humid conditions. The thickness of the ITO thin film was 40 nm, and the thickness of the PET film substrate was 50 µm. The indenter ball used in the wear formation was a 440C stainless steel ball with a load of 0,98 N. The number of slides was 400. As shown in Figure B.2 b), the depth of the wear track was approximately 20 nm to 25 nm. It can be concluded that a vacuum chuck can be used to perform sliding wear formation for fine ceramic thin films deposited onto a thin polymer film or thin glass substrate with a thickness of < 200 µm.



a) Surface image of an ITO-coated PET film after a reciprocating sliding wear formation



b) Depth profile of the wear track for an ITO thin film

Key

X	horizontal position (µm)
Y	height (µm)

Figure B.2 — Results for a reciprocal sliding wear formation of an ITO-coated PET film

Annex C (informative)

Effects of combining thin film materials and indenter materials in sliding wear tests

C.1 Necessity to examine the combination of a thin film material and an indenter ball material

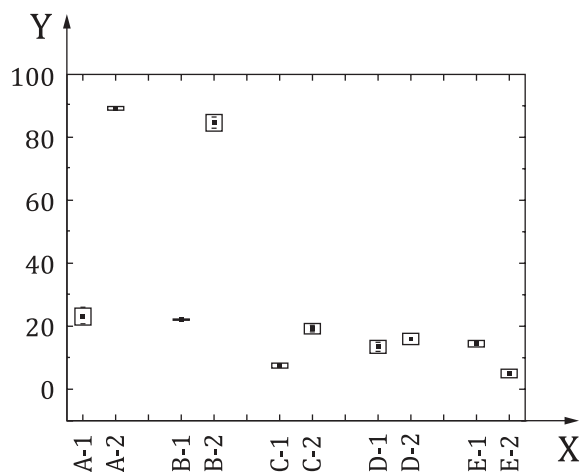
In sliding wear tests for fine ceramic thin films, the combination of a thin film material and an indenter ball material strongly affects the wear behaviour – namely, the wear track formation and friction coefficient – because the wear behaviour, in most cases, results from adhesion wear or abrasive wear in the wear formation for a fine ceramic thin film. In addition, particularly in sliding wear formations performed under dry and humid conditions, the combination of materials affects the test results because the existence of water affects the mode of wear, adhesive or abrasive, thereby resulting in an inverse wear track formation rate and friction coefficient for the two conditions. When fine ceramic thin films are tested by using a fine ceramics indenter ball, the formation of the wear track with a smaller depth is observed sometimes compared to the wear track observed for the cases where they are tested by using a metal indenter ball. Therefore, it is necessary to optimize the combination of the thin film material and the indenter ball material to obtain results that are most relevant to the purpose of the wear test being performed.

C.2 Summary of some results of an investigation into combining a thin film material and an indenter ball material

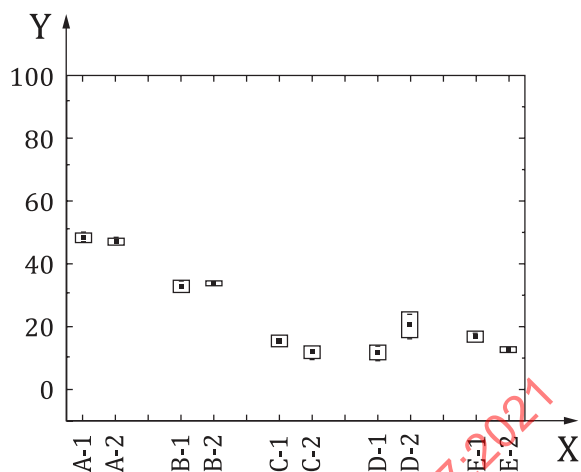
This subclause describes the results obtained by using different combinations of a thin film material and an indenter ball material. [Figure C.1](#) shows the wear track cross-section after rotational sliding tests had been performed using the as-deposited ITO (a), annealed ITO (b), Si_3N_4 (c) and SiO_2 (d) thin films, as described in [Table A.1](#). The indenter balls used in the sliding tests were 304 stainless steel, 440C stainless steel, Al_2O_3 , SiC and Si_3N_4 . The indenter load was set to be 0,25 N and the number of laps was 1 000. In the test performed for ITO thin films without post-deposition annealing, the use of 304 stainless steel or 440C stainless steel as the indenter ball resulted in a large wear cross-section, especially under humid conditions. In contrast, the use of Al_2O_3 , SiC and Si_3N_4 resulted in smaller wear track cross-sections, even under humid conditions. In particular, the use of Si_3N_4 resulted in almost no wear under dry conditions and in a very small wear cross-section, even in humid conditions. In the test performed for ITO thin films with post-deposition annealing, however, the use of 304 stainless steel or 440C stainless steel as the indenter ball resulted in a small wear cross-section, even under humid conditions. In addition, the use of Al_2O_3 , SiC and Si_3N_4 resulted in smaller wear track cross-sections, even under humid conditions. In the test performed for Si_3N_4 or SiO_2 , the use of 304 stainless steel or 440C stainless steel resulted in the same tendency, although in some cases the results were inverted under dry and humid conditions.

[Figure C.2](#) shows the wear track cross-section of as-deposited ITO thin films that were tested using 304 stainless steel, 440C stainless steel, Al_2O_3 , SiC and Si_3N_4 indenter balls. The mode was a reciprocating slide and the number of strokes was 2 000. The indenter load was 0,20 N.

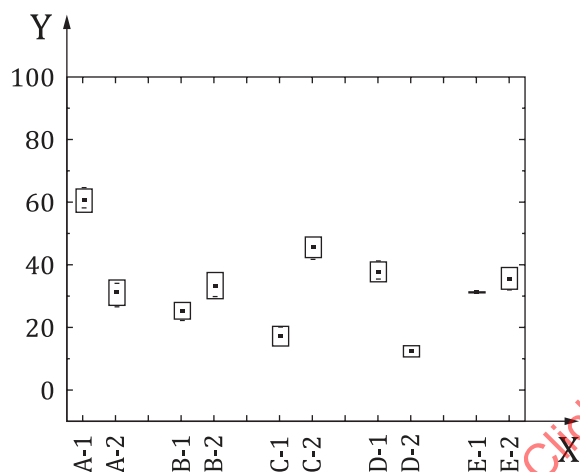
The wear track cross-section was larger for the sliding wear formation performed by using a 304 stainless steel or 440C stainless steel indenter ball than it was for the sliding formation performed by using ceramics indenter balls. In particular, the ITO thin films were not worn in the sliding wear formation performed by using SiC or Si_3N_4 indenter balls. In addition, the tendency for the wear track cross-section to increase for sliding wear formation performed under humid conditions was not exhibited in sliding wear formation performed by using SiC or Si_3N_4 indenter balls.



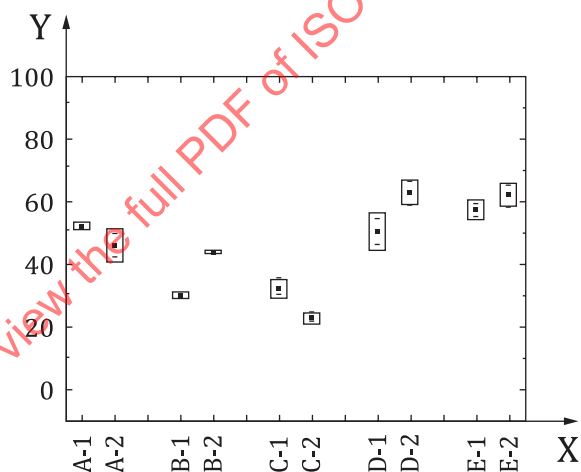
a) Results for an as-deposited ITO (ITO-A) thin film



b) Results for an annealed ITO (ITOB) thin film



c) Results for an Si₃N₄ thin film



d) Results for an SiO₂ thin film

Key

- Y wear track cross-section (μm²)
- average
- max-median-min
- standard deviation
- A-1 10 % RH-304 stainless steel
- A-2 80 % RH-304 stainless steel
- B-1 10 % RH-440C stainless steel
- B-2 80 % RH-440C stainless steel
- C-1 10 % RH-Al₂O₃
- C-2 80 % RH-Al₂O₃
- D-1 10 % RH-SiC

D-2	80 % RH-SiC
E-1	10 % RH-Si ₃ N ₄
E-2	80 % RH-Si ₃ N ₄

NOTE RH represents relative humidity. The tests were performed for a) an as-deposited ITO, b) an annealed ITO, c) a Si₃N₄ thin film and d) a SiO₂ thin film by using 304 stainless steel, 440C stainless steel, Al₂O₃, SiC and Si₃N₄ indenter balls under dry and humid conditions. The indenter load was set to be 0,20 N and the number of rotation lap was 1 000.

Figure C.1 — Wear track cross-section after the rotational sliding wear formations

It is clear from a comparison of the wear track cross-sections shown in [Figures C.1 and C.2](#) that the results are independent of the sliding mode but dependent on the indenter ball materials, implying that the wear results strongly depend on the combination of the thin film material and the indenter ball material.

Changing the combination of a thin film material and an indenter ball material produced inverse results for the tests performed under dry and humid conditions. In the sliding tests performed for SiO₂ thin films, tests performed by using 440C stainless steel yielded a larger cross-section under humid conditions compared to those performed under dry conditions. Furthermore, sliding tests performed by using an Al₂O₃ indenter ball yielded a smaller cross-section than those performed under dry conditions. This behaviour was re-examined in tests performed at other institutes, as shown in [Figure C.3](#).

It should be carefully noted and considered that the results obtained by performing sliding wear formations under dry and humid conditions sometimes yielded inverse results, depending on the combination of the thin film material and the indenter ball material.

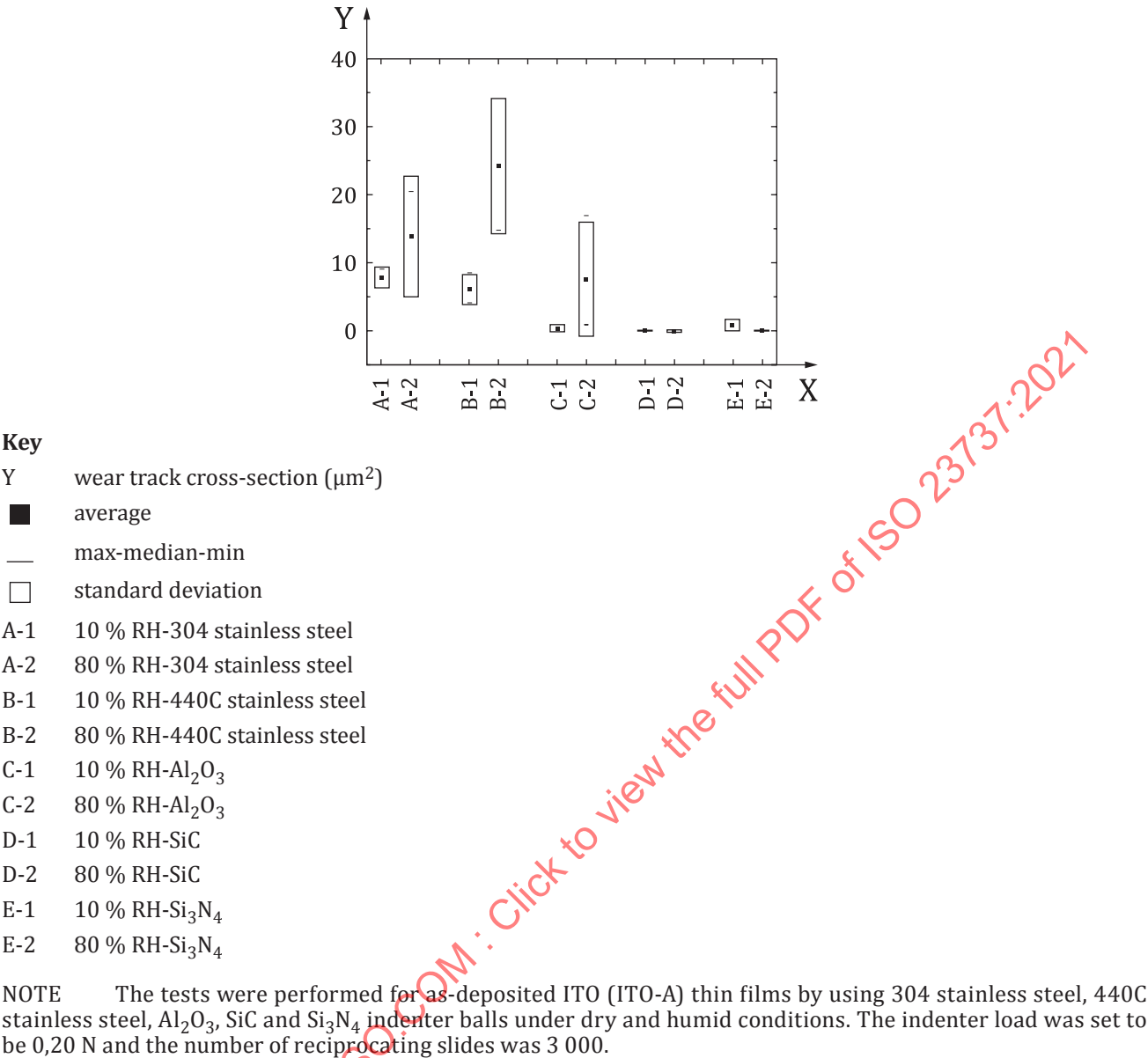
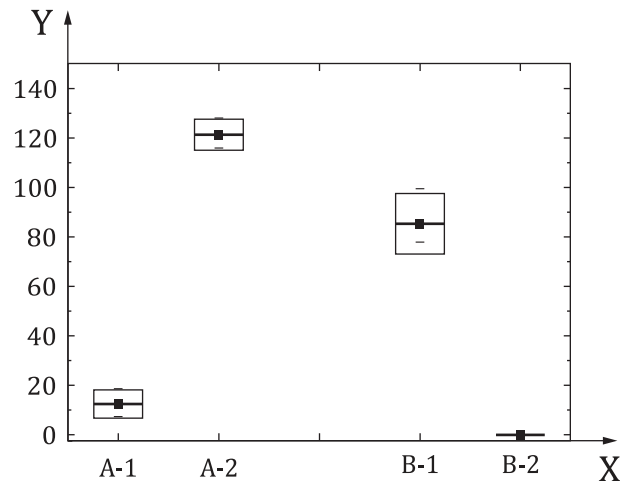


Figure C.2 — Wear track cross-section after the reciprocal sliding wear formations

**Key**Y wear track cross-section (μm^2)

average



max-min



standard deviation

A-1 10 % RH-440C stainless steel

A-2 80 % RH-440C stainless steel

B-1 10 % RH-Al₂O₃B-2 80 % RH-Al₂O₃

NOTE The tests were performed for SiO₂ thin films by using 440C stainless steel and Al₂O₃ indenter balls under dry and humid conditions. The indenter load was set to be 0,20 N and the number of reciprocating slides was 3 000.

Figure C.3 — Wear track cross-section after the reciprocal sliding wear formations

Annex D
(informative)

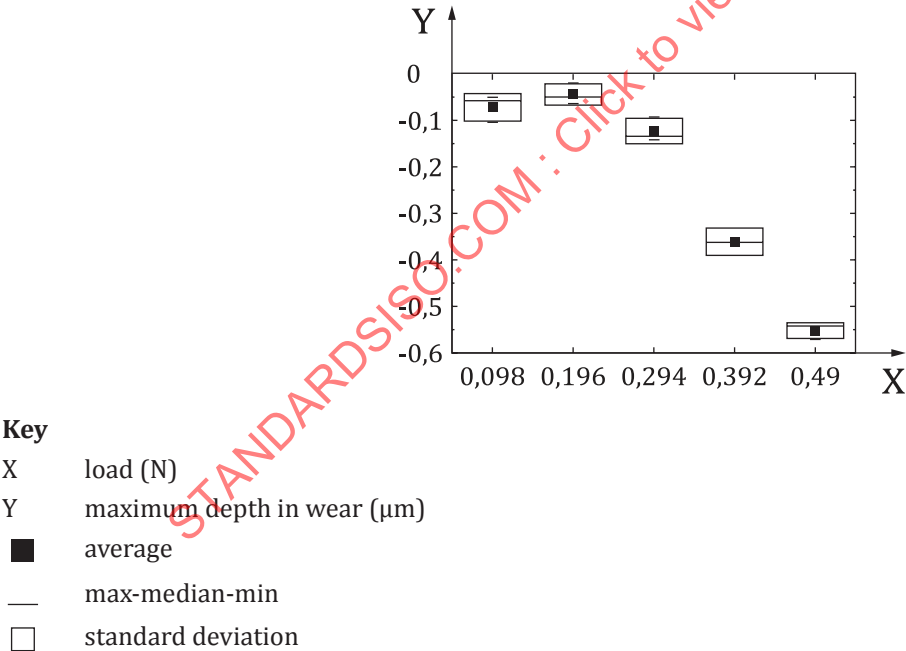
Investigation of optimal load for indenter

D.1 Necessity to optimize the test indenter load

It is necessary to optimize the indenter load to achieve the goal of the wear test. If the indenter load is too high, the wear track penetrates the substrate. In this case, the indenter wears both the thin film and the substrate, thereby providing wear test results that contain those from the substrate, as well. To optimize the indenter load, it is necessary to perform a pre-test to investigate the test load and the number of strokes for which the wear track will not penetrate the substrate. If a certain indenter load that has been determined from the actual application situation needs to be applied, this load should be used in the wear formation.

D.2 Relationship between the indenter load and the maximum wear track depth

Figure D.1 shows an example of the relationship between the indenter load and the maximum wear track depth. The thin film test specimen was as-deposited ITO with a thickness of 300 nm and the indenter ball was 440C stainless steel. The number of reciprocating strokes was 1 000. In the case that was explored, the wear track penetrated the substrate when a load of 0,392 N was applied. Therefore, the indenter load should be $\leq 0,294$ N and the number of strokes should be $< 1\ 000$.



NOTE The thin film test specimen was as-deposited ITO (ITO-A) and the indenter ball was 440C stainless steel. The number of reciprocating slides was 1 000.

Figure D.1 — Relationship between the indenter load and the maximum wear track depth

D.3 Criteria for selecting the indenter load and the number of strokes

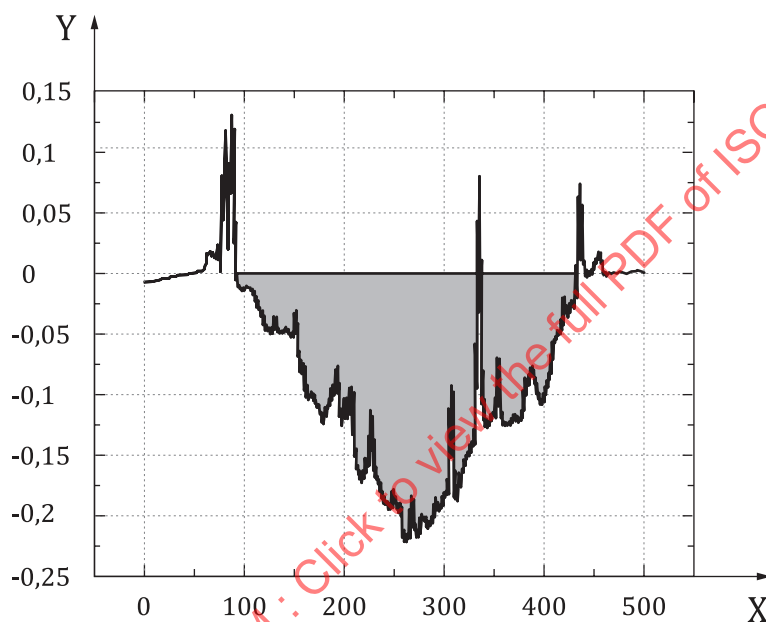
In sliding wear formations for fine ceramic thin films, the maximum wear track depth sometimes penetrated the substrate even if a small indenter load of $< 0,50$ N was applied, particularly for thin films deposited at a low substrate temperature. The indenter load should be sufficiently low to ensure that the wear track remains within the thickness of the thin film with a certain reproducibility. If the purpose of the test is to examine wear under a specified indenter load, this load should be applied.

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Annex E (informative)

Quantification of wear track cross-section area

Figure E.1 shows the method for quantifying a wear cross-section. The quantified wear cross-section is shaded. In this case, the profile should be horizontalized. In most cases, the wear cross-section does not show an identical profile. Based on the actual cross-section, the accuracy can be increased with digital data processing. The ridges formed on the side of the wear track shall be rejected from the measured area. If necessary, cross-sectional images shall be attached to the report.

**Key**

- X horizontal position (μm)
Y wear depth (μm)

Figure E.1 — Quantification of a wear track cross-section