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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

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

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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.

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 °C or lower at an absolute pressure of 101,3 kPa

3.8
dry nitrogen
nitrogen with a dew point of −60 °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 µm 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:

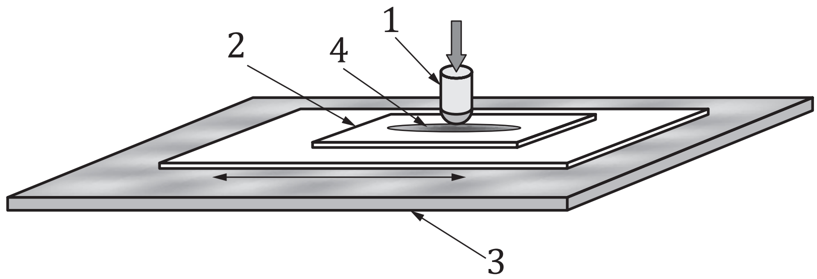
a) testing room temperature: (23 ± 2) °C;

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.

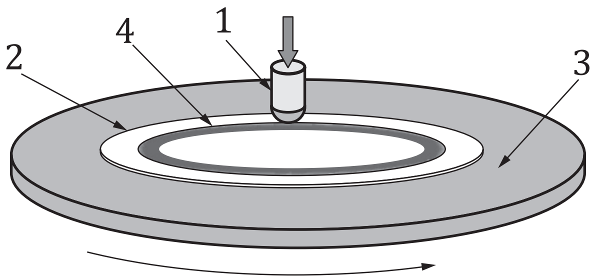
c) The indenter ball holder shall secure the indenter ball reliably against displacement by the frictional force generated at the area of contact between the indenter ball and the test specimen, and shall have high rigidity against induced stress.

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 ± 2) °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 ± 2) °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.

