
**Fine ceramics (advanced ceramics,
advanced technical ceramics) — Test
method for plasma resistance of
ceramic components in semiconductor
manufacturing equipment**

*Céramiques techniques — Méthode d'essai pour déterminer
la résistance au plasma des composants céramiques dans les
équipements de production à semi-conducteurs*

ISO 21859:2019

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Published in Switzerland

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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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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.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for plasma resistance of ceramic components in semiconductor manufacturing equipment

1 Scope

This document specifies a test method for plasma resistance of ceramic components in semiconductor manufacturing equipment. It is applicable to ceramic components of plasma-resistant components in dry etching chambers used in semiconductor manufacturing.

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

ISO 18452, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of thickness of ceramic films by contact-probe profilometer*

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3 Terms and definitions

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

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

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

3.1

plasma resistance

resistance to erosion of the material such as particles which result from corrosion of the materials by erosive plasma source for semiconductor manufacturing

3.2

ceramic component

ceramic part, such as an electrostatic chuck, ring, plate for gas injection, end-point detector, gas injector or viewing port, in a dry etching chamber in semiconductor manufacturing

3.3

erosion depth

difference in height between non-plasma exposure area and plasma exposure area

4 Principle of measurement

This document concerns the measurement of erosion depth after a plasma resistance test and the measurement of surface roughness before and after a plasma resistance test.

An erosion depth shall be evaluated from the profile which is obtained by scanning the surface by using a contact probe profilometer. The profile is in proportion to the difference in height between the parts covered by masking and those not covered by masking.

Surface roughness parameters such as R_a and R_z shall be evaluated before and after the plasma resistance test by using a surface roughness profilometer.

5 Test environment

The measurement of the erosion depth and surface roughness shall be carried out in an environment free from mechanical vibrations that could affect the measurement.

6 Apparatus

6.1 Plasma-etching equipment

The plasma-etching equipment should be a type of reactive ion etching with two parallel electrodes powered by a radio-frequency generator of 13,56 MHz.

6.2 Contact-probe profilometer

The contact-probe profilometer shall be in accordance with ISO 18452.

6.3 Surface roughness profilometer

The surface roughness profilometer shall be in accordance with ISO 3274.

The surface roughness parameters shall be in accordance with ISO 4287.

The contact-probe profilometer and surface roughness profilometer may be the same instrument.

7 Test pieces

7.1 General consideration

The test pieces shall comprise ceramics or a ceramic coating on the substrate.

The test pieces shall have dimensions sufficient to ensure stability on the test piece stage of the plasma-etching equipment, contact-probe profilometer and surface roughness profilometer.

Clean the test piece, by using an appropriate method for the ceramics or ceramic coating, so that the surface of the test piece is free from dust, oil and any other foreign particle.

7.2 Surface conditions

The test piece shall have smooth surface roughness sufficient to measure the erosion depth by using the plasma resistance test.

8 Procedure

8.1 Measurement of surface roughness before a plasma resistance test

Measure the surface roughness of the test piece by using the surface roughness profiler before a plasma resistance test.

8.2 Masking

Cover a part of the test piece surface with ceramic material, such as aluminium oxide.

The ceramic materials for masking shall be fixed so as not to move during the plasma resistance test.

8.3 Plasma resistance test

8.3.1 Setting of test pieces

Set the test pieces on the bottom electrode concentrically.

8.3.2 Test conditions

Use process gases of tetrafluoromethane and oxygen.

The ratio of the flow rate of tetrafluoromethane to the flow rate of oxygen shall be 4 to 1.

Use an incident plasma power of 1 kW and a radio-frequency of 13,56 MHz.

The pressure in the test chamber should be 1 Pa to 100 Pa.

The time of the test shall be enough to evaluate the plasma resistance. The minimum test time should be at least 1 h and the number of testing points should be two or more testing points, such as 5 h and 10 h.

The flow rate of tetrafluoromethane and oxygen should be $6,76 \times 10^{-3} \text{ Pa} \cdot \text{m}^3/\text{s}$, $1,69 \times 10^{-3} \text{ Pa} \cdot \text{m}^3/\text{s}$, respectively.

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8.4 Measurement of erosion depth

Remove the masking materials from the test piece after the plasma resistance test.

Measure the erosion depth of the test piece in the direction perpendicular to the interface between the masked area and the etched surface area by using the contact-probe profilometer.

8.5 Measurement of surface roughness after the plasma resistance test

Measure a surface roughness, such as Ra and Rz , of the test piece in the etched area and the previously masked area by using the surface roughness profiler after the plasma resistance test.

9 Calculation

9.1 Calculation of mean erosion depth

The mean value of the erosion depth is calculated by taking at least five measurements per test piece. The value should be given to three significant figures (validated number).

The mean value of the erosion depth, d_{er} , in μm , is given by [Formula \(1\)](#):

$$d_{er} = \frac{\sum_{i=1}^n d_i}{n} \quad (1)$$

where

d_i is the measurement value;

n is the total number of measurements.

9.2 Calculation of surface roughness

The mean value of surface roughness parameters is calculated by taking at least five measurements per test piece.

The surface roughness of covered parts by masking may be used as the surface roughness before the plasma resistance test.

10 Test report

The test report shall include the following information:

- a) a reference to this document (i.e. ISO 21859);
- b) the name of the testing establishment;
- c) the date of the test, report identification number and signatory;
- d) the size (coating thickness if the test piece is coating material) and shape of the test pieces;
- e) a description of the test material (material type of the substrate and the coating if the test piece is coating material);
- f) plasma resistance test conditions (type of plasma reactor, process gas, gas flow rate, power of incidence plasma, frequency, pressure of the plasma etching chamber, area of the bottom electrode, test time, loading temperature for the heating process);
- g) the method of test piece sampling and preparation;
- h) the test result (erosion depth, surface roughness R_a and R_z before and after plasma etching);
- i) any comments about the test or the test results.

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