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Surface chemical analysis — Determination of surface elemental contamination on silicon wafers by total-reflection X-ray fluorescence (TXRF) spectroscopy

Analyse chimique des surfaces — Détermination de la contamination en éléments à la surface des tranches de silicium par spectroscopie de fluorescence X à réflexion totale

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights (see annex D, note to clause D.2). ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14706 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*.

Annexes A to F of this International Standard are for information only.

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Introduction

This International Standard was prepared for the measurement of surface elemental contamination on silicon wafers on the basis of three existing standards: ASTM F 1526, SEMI Standard M33 and a UCS (Ultra-Clean Society) standard published by the Institute of Basic Semiconductor Technology Development.

TXRF needs reference materials to perform quantitative analyses. Certified reference materials are not available at low densities of 10^{10} atoms/cm^2. Even if they were available, the possibility of contamination from the environment reduces the shelf life of such reference materials.

Therefore, the TXRF reference materials should be prepared and analysed for calibration by each relevant analytical laboratory. Thus, two standards, one for the TXRF measurement procedure and the other for the preparation of reference materials, are necessary. This standard concerns the former part.

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Surface chemical analysis — Determination of surface elemental contamination on silicon wafers by total-reflection X-ray fluorescence (TXRF) spectroscopy

1 Scope

This International Standard specifies a TXRF method for the measurement of the atomic surface density of elemental contamination on chemomechanically polished or epitaxial silicon wafer surfaces.

The method is applicable to:

— elements of atomic number from 16 (S) to 92 (U);

— contamination elements with atomic surface densities from 1×10^{10} atoms/cm^2 to 1×10^{14} atoms/cm^2;

— contamination elements with atomic surface densities from 5×10^{8} atoms/cm^2 to 5×10^{12} atoms/cm^2 using a VPD (vapour-phase decomposition) specimen preparation method (see 3.4).

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2 Normative reference

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The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 14644-1, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness.*

3 Terms and definitions

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

3.1
total reflection
complete reflection of incident radiation at a boundary with a medium in which it travels faster

NOTE The refractive index of incident X-rays impinging on silicon is less than 1. X-rays which are incident on the surface at a small glancing angle are therefore totally reflected from the surface at an angle equal to the glancing angle.

3.2
glancing angle
angle between the specimen surface and the X-rays which impinge on the surface

3.3
critical angle
glancing angle corresponding to the first point of inflection in the plot of the sample matrix X-ray fluorescence against the glancing angle

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3.4
VPD-TXRF method
method in which impurities on the surface are collected by the so-called VPD procedure, i.e. the non-volatile products formed by acid decomposition of the oxide at the wafer surface are collected by a droplet of collecting agent, usually ultra-pure hydrofluoric acid, and dried in a manner which gives the least environmental contamination, the residue from the droplet subsequently being analysed by TXRF

3.5
spurious peaks
detected peaks that do not originate from impurities on the silicon wafer

NOTE Spurious peaks are due to X-rays originating from elements in the detector or the X-ray path. The X-rays are excited by direct scattering or reflection of incident X-rays. This phenomenon leads to an increase in the measurement error. Spurious peaks seriously affect analytical measurements in the contamination range from ca. 10^{10} atoms/cm^2 to ca. 10^{11} atoms/cm^2.

4 Abbreviated terms

FWHM full width at half maximum

RM reference material

SSD solid-state detector

TXRF total-reflection X-ray fluorescence

VPD vapour-phase decomposition

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5 Principle

When a specimen is irradiated with X-rays, fluorescence X-rays at characteristic energies of the elements that constitute the specimen are generated. The intensities of the fluorescence X-rays are proportional to the amounts of each element in the specimen.

Total reflection of the incident X-rays on the specimen reduces the intensity of the scattered X-rays. This allows more intense excitation of the fluorescence X-rays from the surface region, including atoms deposited on the surface of the silicon wafer. Consequently, a spectrum of fluorescence X-rays with a large ratio of signal to background (S/B) and signal to noise (S/N) can be obtained.

The detection limit depends upon the atomic number, the excitation energy, the photon flux, the detector resolution, the energy bandwidth of the excitation X-rays, the instrumental background, the integration time and the blank value. For constant instrumental parameters, the interference-free detection limits vary over two orders of magnitude as a function of the atomic number of the analyte element.

NOTE The depth of measurement may vary with the glancing angle, but in the case of film-type contamination it is usually less than 5 nm. The area of measurement is ca. 10 mm in diameter, though it varies depending on the relative position of the X-ray detector and the specimen. In the case of particulate-type contamination on a clean surface, the yield of fluorescence X-rays varies depending on the sizes, distribution and constituent elements of the particles.

6 Apparatus

6.1 The apparatus for TXRF shall include at least the following components: an X-ray source, a monochromator, a specimen stage capable of movement in three orthogonal directions, an X-ray detector (SSD) and a computerized signal-processing system.

6.2 X-rays which have been monochromatized shall be used as the incident X-rays.

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6.3 The fluorescence X-ray detector shall have sufficient energy resolution to analyse the Mn-K-$L_{II,III}$ line with an FWHM of 200 eV or less.

6.4 The specimen stage, which sets the glancing angle, shall be controlled to a reproducibility within ± 0,17 mrad (0,01°) in the range between 0 mrad (0°) and 8,7 mrad (0,5°).

6.5 The atmosphere in the specimen chamber shall be able to be brought to a reduced pressure or replaced with helium gas or nitrogen gas, as required.

7 Environment for specimen preparation and measurement

7.1 The local environment (i.e. airborne particles, temperature, humidity) for specimen preparation and measurement shall be equal to or better than ISO Class 4 (see ISO 14644-1).

NOTE The unwanted deposition of airborne particles which are composed of the elements that are being measured will cause an increase in the error of measurement.

7.2 The mechanical vibration at the location where the apparatus is installed shall be as small as possible and shall not be greater than 5×10^{-3} m/s^2 (0,5 Gal).

NOTE The mechanical vibration will degrade the energy resolution of the detection system, which will, in turn, degrade the detection limits and peak deconvolution.

8 Calibration reference materials

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8.1 Calibration reference materials (RMs) used to establish a reliable calibration procedure shall consist of an RM on which known amounts of impurities have been deposited and a blank RM used to determine the level of contamination of the RM (see annex A).

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8.2 The RMs shall be prepared from a chemomechanically polished wafer with a certain quantity of Ni or Fe uniformly deposited on its surface as the RM element. The atomic surface density of the RM element shall be ca. 1×10^{12} atoms/cm^2 (see annex C).

8.3 How the RM element is located on the RM surface shall be verified by an anglescan (see annex E).

8.4 The amount of RM element deposited on the surface of the wafer shall be determined by a reliable quantitative method of analysis.

8.5 The blank RM shall be a chemomechanically polished or epitaxial wafer. The impurity of the surface region of the blank RM shall be below the detection limit. The crystallographic orientation of the blank RM shall be the same as that of the RM.

8.6 The RM and the blank RM shall be stored in the same container.

9 Safety

This test method uses X-ray radiation. Consequently, it is absolutely essential to avoid exposing any part of the body to the X-rays produced by the apparatus. It is especially important to keep hands and fingers out of the path of the X-rays and to protect the eyes from scattered secondary X-ray radiation. Each country has its own safety regulations and requirements concerning X-rays. These shall be observed.

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10 Measurement procedure

10.1 Preparation for measurement

10.1.1 All specimens shall be chemomechanically polished or epitaxial wafers.

10.1.2 For the VPD-TXRF method, the impurities on the surface are collected by VPD, i.e. acid decomposition in a droplet which is then dried in a manner that produces the least environmental contamination (see annex D).

10.1.3 For a series of measurements, including the calibration measurements, the crystal orientation of the specimen on the specimen stage shall be the same for each specimen.

10.1.4 Set the glancing angle at between 25 % and 75 % of the critical angle.

NOTE Due to the physical form of particulate-type contamination, an angle that is too low will cause a larger error.

NOTE The critical angle is a function of the incident X-ray energy. It is 3,20 mrad (0,18°) for 9,67 keV (W-L_{II}-M_{IV}), 1,78 mrad (0,10°) for 17,44 keV (Mo-K-$L_{II,III}$) and 2,72 mrad (0,16°) for 11,4 keV (Au-L_{II}-M_{IV}).

10.1.5 Set the following parameters as specified:

a) For a rotating anode, the excitation voltage of the X-ray source shall be 30 kV or more, with the excitation current set to 200 mA or more and the integration time set to 500 s or more.

b) For a sealed Mo or W anode X-ray tube, the excitation voltage of the X-ray source shall be 30 kV or more, with the excitation current set to 40 mA or more and the integration time set to 500 s or more.

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c) If the X-ray intensity at the detector is too high for the detection system, the excitation current shall be adjusted to a lower value to give an appropriate count rate.

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10.1.6 Move the centre of the specimen under the centre of the detector. While rotating the specimen, generate and measure fluorescence X-rays to find the azimuth with the least spurious peaks. When possible, the measurement hereafter shall be conducted at the same azimuth.

If the apparatus does not allow the best-fit azimuth to be set at off-centre positions so as to avoid spurious peaks, care shall be taken when evaluating the surface-mapping data.

10.2 Preparing a calibration curve

10.2.1 Measure the blank RM and determine the integrated intensity of the fluorescence X-rays generated by the blank RM.

10.2.2 Measure the RM under the same conditions as specified in 10.2.1. In the case of the VPD-TXRF method, place the residue from the RM under the centre of the detector.

10.2.3 Verify that the measured value of the integrated intensity of the X-rays generated by the blank RM is less than 10 % of the integrated intensity from the RM.

If the integrated intensity from the blank RM is 10 % or more of that from the RM, discard both the RM and the blank RM and prepare a new set comprising an RM and blank RM.

Carry out this check at appropriate intervals.

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10.2.4 Determine the integrated intensity of the X-rays generated by the RM element by means of procedure a) or b) specified below:

a) Smooth the measured numerical values by digital processing.

   Determine the integrated intensity of the X-rays by subtracting the background from the measured numerical values.

b) Determine the Gaussian function that best fits the measured numerical values. Then determine the integrated intensity from the peak height and the half-width of the Gaussian function.

10.2.5 Obtain a calibration curve (a plot of the atomic surface density versus the integrated X-ray intensity from the RM element). The curve shall pass through the origin.

10.3 Measurement of a test specimen

10.3.1 Measure the test specimen under the same conditions as specified in 10.1. If using the VPD-TXRF method, place the residue from the test specimen under the centre of the detector.

10.3.2 Determine the integrated intensity of the X-rays generated by contamination elements in the same manner as specified in 10.2.

When two or more fluorescence X-ray lines overlap, use the method of deconvolution to obtain the integrated intensity of the X-rays for the subject element.

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NOTE 1 The repeatability and reproducibility of the measurement for the subject element will vary with the kind of X-ray used.

NOTE 2 Deviation of the glancing angle of the incident X-rays from the set value will increase the measurement error.

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NOTE 3 Greater surface roughness will increase the measurement error.

NOTE 4 The values obtained from VPD residues will depend greatly on the physical form of the residue and the elements contained in the residue.

11 Expression of results

11.1 Method of calculation

By using equations (1) and (2), calculate the atomic surface density C_m for each of the contamination elements from the results obtained in clause 10.

$$K = \frac{C_\mathrm{S}}{I_\mathrm{S}} \quad (1)$$

$$C_\mathrm{m} = K \times \frac{I_\mathrm{m}}{S_\mathrm{R}} \quad (2)$$

where

K is the slope of the calibration curve obtained in 10.2.5;

C_S is the atomic surface density of the RM element, in atoms/cm^2;

I_S is the integrated intensity of the fluorescence X-rays from the RM element, in counts per second (cps);

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C_m is the atomic surface density of the contamination element on the test specimen, in atoms/cm^2;

I_m is the integrated intensity of the fluorescence X-rays from the contamination element on the test specimen, in cps;

S_R is the relative sensitivity factor, which corrects for the difference in sensitivity for each element.

NOTE In order to determine values of the relative sensitivity factor S_R for other elements, measurements are often made on two or more specimens with differing contents of these elements. The results can then be used to prepare calibration curves other than those obtained using theoretical relative sensitivity factors. The relative sensitivity factor can be determined in terms of these curves using the RM element or can be calculated as shown in annex B.

11.2 Blank correction

For measurements below 10^{11} atoms/cm^2, where the instrumental blank is not negligible, the atomic surface density C_m shall be corrected by subtracting the atomic surface density C_0 of a fresh contamination-free specimen, with the same crystal orientation, measured under the same conditions as specified in 10.1 and 10.2 and calculated as described in 11.1.

12 Precision

An international inter-laboratory test programme based on the method specified in this International Standard was carried out by 15 laboratories from Japan, Europe and USA. Four test specimens and one RM were distributed as one set. 17 sets of measurements were obtained from the 15 laboratories. Repeatability and reproducibility were calculated in accordance with the principles of ISO 5725-2 [1]. A statistical report of the inter-laboratory test is given in annex F.

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13 Test report

The test report shall include the following items:

a) specimen identification;

b) the kind(s) of X-ray source used, e.g. rotating-anode W-tube;

c) the excitation X-rays used, e.g. W-L_{II}-M_{IV};

d) the voltage applied to the X-ray source, e.g. 30 kV;

e) the current applied to the X-ray source, e.g. 200 mA;

f) the glancing angle used, e.g. 1,8 mrad (0,10°);

g) the integration time, e.g. 500 s;

h) the method used to prepare the RM, e.g. SC1 dip method (see annex A);

i) the atomic surface density of the RM element (Ni or Fe), e.g. Ni $1{,}05 \times 10^{12}$ atoms/cm^2;

j) the measurement location on the test specimen, e.g. centre of wafer;

k) the calibration method used, i.e. procedure a) or b) in 10.2.4;

l) the name(s) of the element(s) on the test specimen and the atomic surface density of each.

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