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

ICS 71.040.40

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal of establishing a Japanese Industrial Standard from The Foundation of Osaka Science & Technology Center (OSTEC)/ Japanese Standards Association (JSA), with a draft of Industrial Standard based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

This Standard has been made based on **ISO 14706 : 2000** *Surface chemical analysis—Determination of surface elemental contamination on silicon wafers by total-reflection X-ray fluorescence (TXRF) spectroscopy* for the purposes of making it easier to compare this Standard with International Standard; to prepare Japanese Industrial Standard conforming with International Standard; and to propose a draft of an International Standard which is based on Japanese Industrial Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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

Introduction This Japanese Industrial Standard has been prepared based on the first edition of **ISO 14706** *Surface chemical analysis—Determination of surface elemental contamination on silicon wafers by total-reflection X-ray fluorescence (TXRF) spectroscopy* published in 2000 without modifying the technical contents of the original International Standard.

The portions given dotted underlines are the matters not stated in the original International Standard.

The numbers in the brackets [] following the terms and writing in the forward, contents, text and annex indicate the number of the bibliography.

ISO 14706 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/cm². 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.

1 Scope This 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/cm² to 1×10^{14} atoms/cm²;
- contamination elements with atomic surface densities from 5×10^8 atoms/cm² to 5×10^{12} atoms/cm² using a VPD (vapour-phase decomposition) specimen preparation method (see **3.4**).

NOTE : The International Standard corresponding to this Standard is as follows.

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21**.

ISO 14706 : 2000 *Surface chemical analysis—Determination of surface elemental contamination on silicon wafers by total-reflection X-ray fluorescence (TXRF) spectroscopy* (IDT)

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS B 9920 *Classification of air cleanliness for cleanrooms*

NOTE : **ISO 14644-1** : 1999 *Cleanrooms and associated controlled environments—Part 1 : Classification of air cleanliness* is identical with the said standard.

JIS Z 8402-2 *Accuracy (trueness and precision) of measurement methods and results—Part 2 : Basic method for the determination of repeatability and reproducibility of a standard measurement method*

NOTE : **ISO 5725-2** : 1994 *Accuracy (trueness and precision) of measurement methods and results—Part 2 : Basic method for the determination of repeatability and reproducibility of a standard measurement method* is identical with the said standard.

3 Definitions For the purposes of this Standard, the following 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-ray 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

Information : The word of glancing angle is a correct term, however the word of incident angle is also used generally.

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

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

Information : It is also called as an apparent peak of impurities.

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/cm² to ca. 10^{11} atoms/cm².

4 Abbreviated terms

FWHM full width at half maximum

RM reference material

SSD solid-state detector

TXRF total-reflection X-ray fluorescence

Information : TXRF is generally used as the abbreviation of total-reflection X-ray fluorescence spectroscopy.

VPD vapour-phase decomposition

Information : It was originally the vapour-phase decomposition method with using acidic vapour, however the method, in which the residual non-volatile component is recovered as dissolving directly oxide film with solution drop without using vapour, is called as VPD in wide sense.

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

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.

Information : Mn-K-L_{II,III} is also called as MnK_α.

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 **JIS B 9920** Class 4.

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/s² (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

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).

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/cm² (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.

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.

NOTES 1 Due to the physical form of particulate-type contamination, an angle that is too low will cause a larger error.

2 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 17.4 keV (Mo-K-L_{II,III}) and 2.72 mrad (0.16°) for 11.4 keV (Au-L_{II}-M_{IV}).

Information : W-L_{II}-M_{IV}, Mo-K-L_{II,III} and Au-L_{II}-M_{IV} are also called as WL_{β1}, MoK_α and AuL_{β1} respectively.

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.

- 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.

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 position 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-ray 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.

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 measure 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.