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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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Foreword

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*.

This second edition cancels and replaces the first edition (ISO 14706:2000), which has been technically revised.

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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 are to 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 International 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 the following:

— 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).

2 Normative reference

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The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

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

3.1
total reflection
complete reflection of glancing X-ray radiation into a medium having larger X-ray optical density value at a boundary between two media

Note 1 to entry: The refractive index of silicon for X-rays 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 plane and the virtual plane containing incident X-rays which impinge on the sample 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 1 to entry: Spurious peaks are due to fluorescent X-rays originating from elements in the detector or the X-ray path. The fluorescent 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

SDD silicon drift 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 a penetration depth of the incident X-rays in the specimen. This allows more selective 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 and energy-dependent detection efficiency, the energy bandwidth of the excitation X-rays, the specimen-related shape and statistics of TXRF spectral background, the instrument-related noise magnitude, the integration time, and the accuracy of calibration of the RM 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 can vary with the glancing angle, but in the case of film-type contamination it is usually less than 5 nm. The area of measurement consists of a circle of 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.

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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 or SDD); and a computerized signal-processing system.

6.2 X-rays 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.

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 in accordance with 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.

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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).

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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 calibration 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/cm^2 to 1×10^{13} 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 magnitude of contamination of the surface region of the blank RM shall be below the detection limit for specified elements. 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.

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