

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

ICS 71.040.40

National foreword

This British Standard reproduces verbatim ISO 14706:2000 and implements it as the UK national standard.

The UK participation in its preparation was entrusted to Technical Committee CII/60, Surface chemical analysis, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

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This British Standard, having been prepared under the direction of the Sector Committee for Materials and chemicals, was published under the authority of the Standards Committee and comes into effect on 15 May 2001

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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

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.

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/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.

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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/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).

2 Normative reference

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

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/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

VPD vapour-phase decomposition

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.