

Surface chemical analysis — Chemical methods for the collection of elements from the surface of silicon-wafer working reference materials and their determination by total-reflection X-ray fluorescence (TXRF) spectroscopy

ICS 71.040.40

National foreword

This British Standard is the UK implementation of ISO 17331:2004+A1:2010. It supersedes BS ISO 17331:2004 which is withdrawn.

The start and finish of text introduced or altered by amendment is indicated in the text by tags. Tags indicating changes to ISO text carry the number of the ISO amendment. For example, text altered by ISO amendment 1 is indicated by **A1** **A1**.

The UK participation in its preparation was entrusted to Technical Committee CII/60, Surface chemical analysis.

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

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fluorescence (TXRF) spectroscopy**

*Analyse chimique des surfaces — Méthodes chimiques pour collecter
les éléments analysés de tranches de silicium comme matériaux de
référence pour l'analyse par spectroscopie de fluorescence X en
réflexion totale (TXRF)*



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Foreword

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The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights (see Note to 10.1). ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17331 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*.

Introduction

The measurement of metal impurities on the surface of silicon wafers by total-reflection X-ray fluorescence spectroscopy (TXRF) is commonly accepted by the semiconductor industry.

ISO 14706 specifies a TXRF method for the measurement of metal impurities on a silicon-wafer surface with atomic surface densities from 1×10^{10} atoms/cm² to 1×10^{14} atoms/cm², also a vapour-phase decomposition (VPD) method for atomic surface densities from 5×10^8 atoms/cm² to 5×10^{12} atoms/cm².

In ultra-large-scale integrated circuit (ULSI) manufacturing, the measurement of very-low-level metal impurities on the surface of a silicon wafer, less than 10^{10} atoms/cm², is currently required.

Reference materials with low-level metal impurities are important in TXRF analysis (see ISO 14706), but the reference materials have a limited shelf life, especially as regards surface contamination. As a result, preparation methods for working reference materials need to be standardized.

This test method allows the calculation of metal impurities on the surface of reference and test wafers. Interlaboratory trials in nine laboratories in different countries indicated that this method has good reproducibility and repeatability.

A1 The International Organization for Standardization (ISO) draws attention to the fact that it is claimed that compliance with this document may involve the use of patents concerning the collection of elements from the materials given in 10.2 and 10.3.

ISO takes no position concerning the evidence, validity and scope of these patent rights.

The holders of these patent rights have assured ISO that they are willing to negotiate licences under reasonable and non-discriminatory terms and conditions with applicants throughout the world. In this respect, the statements of the holders of these patent rights are registered with ISO. Information may be obtained from:

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Surface chemical analysis — Chemical methods for the collection of elements from the surface of silicon-wafer working reference materials and their determination by total-reflection X-ray fluorescence (TXRF) spectroscopy

1 Scope

[A1] This International Standard specifies chemical methods for the collection of iron and/or nickel from the surface of silicon-wafer working reference materials by the vapour-phase decomposition method or the direct acid droplet decomposition method and their determination by total-reflection X-ray fluorescence (TXRF) spectroscopy. **[A1]**

NOTE The determination of the elements collected may be carried out by graphite-furnace atomic-absorption spectroscopy or inductively coupled plasma mass spectrometry instead of by total-reflection X-ray fluorescence spectroscopy.

This International Standard applies to iron and/or nickel atomic surface densities from 6×10^9 atoms/cm² to 5×10^{11} atoms/cm².

2 Normative references

The following referenced documents are indispensable for the application 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 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*

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*

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

3 Terms and definitions

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

3.1

working reference material

silicon wafer used for the calibration specified in ISO 14706

3.2

calibration specimen

silicon wafer with a known amount of iron and/or nickel in a dried residue, used for calibration purposes