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Surface chemical analysis — Proposed procedure for certifying the retained areic dose in a working reference material produced by ion implantation

Analyse chimique des surfaces — Mode opératoire proposé pour certifier la dose aréique retenue dans un matériau de référence de travail produit par implantation d'ions

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Foreword

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In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 16268 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 2, *General procedures*.

Introduction

This Technical Report brings together experience to provide a proposed procedure, untested as a full procedure, to address the general problem of how to obtain a certified working reference material (WoRM) for the quantitative surface chemical analysis of a given solid material available in wafer (disc) form. The WoRM discussed here is essentially an ion-implanted wafer, where the virgin wafer — chosen or prepared by the analyst — has been ion-implanted with, typically, one isotope of a chemical element (henceforth referred to as the analyte) of an atomic number larger than that of silicon. This WoRM is certified by the proposed procedure for the areic dose of the analyte retained.

The retained areic dose of the ion-implanted analyte in the WoRM wafer is certified by comparative measurement against the retained areic dose of the same analyte in an ion-implanted silicon wafer having the status of a (preferably certified) secondary reference material (SeRM). The comparative measurement is performed in a two-step process in which an intermediary third reference material and two measurement techniques [wavelength-dispersive X-ray fluorescence spectrometry (WD/XFS) and ion-implantation dosimetry] are used. The intermediary reference material, referred to as a transfer reference material (TrRM), is also an ion-implanted silicon wafer and is a (non-identical) implantation twin of the WoRM (i.e. it is co-produced with the WoRM but differs in wafer type and retained areic dose). Its function is, firstly, to avoid possible secondary-excitation effects in a direct WD/XFS measurement on the WoRM and, secondly, to allow the WoRM to be certified also for retained areic dose levels far below the measuring range of WD/XFS.

This certification of the WoRM is part of a new concept and procedure for characterization of reference materials. In this concept, the WoRM, TrRM and SeRM have their places in a chain of reference materials and a sequence of certifications. The SeRM is at the interface between the area of responsibility of the analyst and that of a commercial supplier of reference materials. This Technical Report describes the part of the procedure within the area of responsibility of the analyst and is based on the assumption that a suitable SeRM is obtainable. When an SeRM is available, the analyst must also have access to a suitable ion implanter and to a suitable wavelength-dispersive X-ray fluorescence spectrometer for comparative measurement of retained areic doses.

The wafer format requirement of the WoRMs implies a particular suitability for the analysis of semiconductor materials, although it is by no means restricted to this application. A restriction exists, however, in the choice of surface-analytical technique. Although specimen and WoRM may be identical in analyte and host matrix, the analyte may be present in a different chemical state and a different depth distribution. Meaningful results from referencing to the WoRM can then be obtained only if the chosen surface-analytical technique is insensitive to the chemical state of the analyte and if the technique allows corrections for different depth distributions. This problem is addressed with special reference to analysis by secondary-ion mass spectrometry. With an appropriate choice of surface-analytical technique, the WoRMs can be used for quantitative measurement of homogeneous, ion-implanted, diffused and layered depth distributions of the analyte.

This Technical Report is essentially based on Reference [1]. This work has also been a project (Technical Working Area 2/Project 5) within the international Versailles Project on Advanced Materials and Standards (VAMAS)[2].

Surface chemical analysis — Proposed procedure for certifying the retained areic dose in a working reference material produced by ion implantation

1 Scope

This Technical Report specifies a procedure for the certification of the areic dose of an ion-implanted analyte element of atomic number larger than that of silicon retained in a working reference material (WoRM) intended for surface-analytical use. The WoRM is in the form of a polished (or similarly smooth-faced) wafer (also referred to as the host), of uniform composition and nominal diameter 50 mm or more, that has been ion-implanted with nominally one isotope of a chemical element (also referred to as the analyte), not already present in the host, to a nominal areic dose normally within the range 10^{16} atoms/cm^2 to 10^{13} atoms/cm^2 (i.e. the range of primary interest in semiconductor technology). The areic dose of the ion-implanted analyte retained in the WoRM wafer is certified against the areic dose of the same analyte retained in an ion-implanted silicon wafer having the status of a (preferably certified) secondary reference material (SeRM).

Information is provided on the concept and the procedure for certification of the WoRM. There is also a description of the requirements for the reference materials, the comparative measurements and the actual certification. Supporting information on ion implantation, ion-implantation dosimetry, wavelength-dispersive X-ray fluorescence spectroscopy and non-certified substitutes for unobtainable SeRMs is provided in Annexes A to D. Sources and magnitudes of uncertainties arising in the certification process are detailed in Annex E.

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 18115, *Surface chemical analysis — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 and the following apply.

3.1
certification
⟨of a reference material, by a procedure⟩ act of establishing the traceability of a property value to an accurate realization of the unit in which the property value is expressed, where the certified value is accompanied by an uncertainty value at a stated level of confidence

NOTE The term is used for both "the action of making certain" (i.e. certification by a procedure) and "the issuing of a certificate" stating what has been certified by the procedure.

3.2
lower critical energy
kinetic energy of an ion beam below which the backscattering of perpendicularly incident ions exceeds a specified percentage of the received areic dose

3.3
definitive method
⟨of referencing⟩ method based on a valid, well-described theoretical foundation ensuring negligible systematic errors relative to end-user requirements, allowing a property to be measured either directly in terms of basic units of measurement or in terms closely related to the base units through physical or chemical theory expressed in exact mathematical equations

NOTE A definitive method is a special method of reference (see ISO Guide 30[9]) particularly suitable for the certification of primary reference materials by “allowing the property in question to be either measured directly in terms of basic units of measurement or in terms closely related to the base units”. An example thereof would be the vapour deposition of a high-purity element on a wafer and the measurement of the deposit by direct weighing.

3.4
areic dose
dose density (deprecated)
quotient of dN by dA, where dN is the number of particles of a specified type from a mono-energetic, mass-analysed, quasi-parallel particle beam incident on a solid and suffering a specified fate on or after passing through a geometric surface area dA

NOTE 1 The particles may be monoatomic or multiatomic. The chemical type, isotopic type and charge state of the particles before incidence on the solid have to be specified.

NOTE 2 The geometric surface area refers to the areal measure of the projection of the usually micro-rough surface onto an ideal plane parallel to that surface of the solid.

NOTE 3 Areic dose is a generic term requiring further specification concerning the temporary or permanent fate of the particles before numeric values can be assigned. The fate of the particles refers to states of the particles prior to, during or after encounter with the solid, such as incidence on, transmission through, backscattering from, stopping within, re-emission by sputtering from, or retention in the solid.

3.5
implanted areic dose
D^{imp}
quotient of dN^{imp} by dA, where dN^{imp} is the number of particles of a specified type from a mono-energetic, mass-analysed, quasi-parallel particle beam incident on a solid within a geometric surface area dA and captured within the solid

$$D^{\text{imp}} = \mathrm{d}N^{\text{imp}}/\mathrm{d}A$$

NOTE 1 The particles may be monoatomic or multiatomic. The chemical type, isotopic type and charge state of the particles before incidence on the solid have to be specified.

NOTE 2 The geometric surface area refers to the areal measure of the projection of the usually micro-rough surface onto an ideal plane parallel to that surface of the solid.

NOTE 3 The implanted areic dose is smaller than the received areic dose if some of the particles incident on the solid are transmitted through or backscattered from the solid.

3.6
lower critical value of areic dose
⟨for referencing one reference material with respect to another by means of wavelength-dispersive X-ray fluorescence spectrometry⟩ minimum value of the retained areic dose necessary for the repeatability of a specified measurement of this dose by this method to meet a given requirement

3.7
nominal areic dose
D^{nom}
nominal (approximate and averaged) value of the received areic dose, obtained from the quotient of the particle equivalent of the beam current integral over time and the surface area over which the beam is scanned with the best lateral uniformity possible in a given ion implanter

NOTE An analyte ion beam is always contaminated to some, although sometimes negligible, extent by analyte neutrals as well as by non-analyte charged particles. Also, ion dosimetry may be flawed. Therefore, the beam current integral over time is normally only an approximate measure of the number of analyte particles received. Also, the beam scanning may not be entirely uniform and thus the nominal areic dose is an approximate average measure of the received areic dose.

3.8
received areic dose
dose density (deprecated)
D^{rec}
quotient of $\mathrm{d}N^{\mathrm{rec}}$ by $\mathrm{d}A$, where $\mathrm{d}N^{\mathrm{rec}}$ is the number of particles of a specified type from a mono-energetic, mass-analysed, quasi-parallel particle beam incident on a solid within a geometric surface area $\mathrm{d}A$

$$D^{\mathrm{rec}} = \mathrm{d}N^{\mathrm{rec}}/\mathrm{d}A$$

NOTE 1 The particles may be monoatomic or multiatomic. The chemical type, isotopic type and charge state of the particles before incidence on the solid have to be specified.

NOTE 2 The geometric surface area refers to the areal measure of the projection of the usually micro-rough surface onto an ideal plane parallel to that surface of the solid.

NOTE 3 The nominal areic dose is often wrongly substituted for the received areic dose and even for the retained areic dose.

3.9
retained areic dose
D^{ret}
quotient of $\mathrm{d}N^{\mathrm{ret}}$ by $\mathrm{d}A$, where $\mathrm{d}N^{\mathrm{ret}}$ is the number of particles of a specified type from a mono-energetic, mass-analysed, quasi-parallel particle beam incident on a solid within a geometric surface area $\mathrm{d}A$ and permanently retained within the solid

$$D^{\mathrm{ret}} = \mathrm{d}N^{\mathrm{ret}}/\mathrm{d}A$$

NOTE 1 The particles may be monoatomic or multiatomic. The chemical type, isotopic type, and charge state of the particles before incidence on the solid have to be specified.

NOTE 2 The geometric surface area refers to the areal measure of the projection of the usually micro-rough surface onto an ideal plane parallel to that surface of the solid.

NOTE 3 The retained areic dose is smaller than the implanted areic dose if some of the implanted particles are re-emitted by sputtering from the solid. The amount by which the retained areic dose is less than the implanted areic dose increases with increasing implanted areic dose.

3.10
upper critical value of areic dose
⟨for referencing one reference material with respect to another by means of ion-implanter dosimetry⟩ value of the implanted areic dose at which the deviation of the retained areic dose from the implanted areic dose reaches a given small percentage

NOTE The upper critical value of the areic dose is the highest value of the implanted areic dose at which the conditions of quantitative ion implantation are still met.

3.11
implantation conditions
energy, composition (inclusive of charge states), current, diameter, angle of incidence and scanning parameters of the ion beam at the target station, in addition to the target wafer, implanted area and nominal areic dose (and, by implication, implantation time)

3.12
lower critical implantation time
time required to complete one hundred identical ion-beam scan patterns

3.13
implanter operating conditions
ion-implanter settings that influence the energy, composition (inclusive of charge states), current, diameter, angle of incidence and scanning parameters of the ion beam at the target station on the implantation end of the ion implanter

NOTE The residual pressure in the ion implanter can have a significant influence on the ion-beam composition.

3.14
ion implantation
process whereby, in a vacuum environment, a beam of ions of a specified type and of sufficient kinetic energy is caused to penetrate a solid for the purpose of being retained therein

3.15
quantitative ion implantation
dose-limited ion implantation under conditions where, within experimental error, the implanted areic dose equals the received areic dose, and the deviation of the retained areic dose from the implanted areic dose remains below a given small percentage

3.16
overscan arrangement
target station design in an ion implanter in which one or more Faraday cups are situated at the perimeter of the target wafer such that the aperture of each cup is in the same plane as the surface of the target wafer, and the ion beam is scanned in a laterally uniform mode at right angles across the target wafer and the Faraday cup(s)

3.17
reference material
material or substance one or more of whose properties are sufficiently well established to be used for the calibration of an apparatus, for the assessment of a measurement method or for assigning values to materials

NOTE This definition deviates from that in ISO Guide 30:1992[9] by omission of the words “homogeneous and” after “sufficiently” since the ISO Guide 30 definition omitted to consider ion-implanted materials which, by nature, are inhomogeneous in the depth dimension.

3.18
certified reference material
reference material (as defined in 3.17), accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property units are expressed, and for which each certified value is accompanied by an uncertainty value at a stated level of confidence

NOTE For ion-implanted reference materials, the certified property values must include the retained areic dose averaged over the area of implantation, the point-to-point variation of the retained areic dose, the size and exact location of the area of implantation, the kinetic energy of implantation, and preferably also a graphical or mathematical representation of the depth distribution.

3.19
primary (ion-implanted) reference material
certified reference material, consisting of a high-purity silicon wafer ion-implanted with the analyte, that all other ion-implanted reference materials are referenced against (directly or indirectly), the certified property being the retained areic dose (inclusive of the lateral uniformity thereof) determined by a definitive method (as defined in 3.3)

NOTE The primary reference material is used solely for purposes of certification of secondary reference materials that are to be issued to analysts.

3.20
secondary (ion-implanted) reference material
ion-implanted certified reference material, nominally identical to the primary reference material in material and areic dose, serving as an intermediary between a primary reference material and a working reference material, the certified property being the retained areic dose (inclusive of the lateral uniformity thereof) determined by a comparative measurement against the primary reference material

3.21
transfer (ion-implanted) reference material
ion-implanted certified reference material, nominally identical to the secondary reference material in material and areic dose, co-produced with the working reference material and serving as an intermediary between a secondary reference material and a working reference material, the certified property being the retained areic dose (inclusive of the lateral uniformity thereof) determined by a comparative measurement against a secondary reference material

NOTE Each working reference material is paired with a transfer reference material that is ion-implanted in the same implanter under invariant (and hence identical) implanter operating conditions.

3.22
working (ion-implanted) reference material
certified reference material, consisting of a wafer of a composition specified by the analyst, ion-implanted with the analyte for direct use in a surface analysis, the certified property being the retained areic dose (inclusive of the lateral uniformity thereof) determined by a comparative measurement against a secondary reference material via a transfer reference material

3.23
target wafer
host wafer
virgin wafer subjected to ion implantation

4 Symbols and abbreviated terms

CRM certified reference material

D areic dose

D^{imp} implanted areic dose

D^{nom} nominal areic dose

$D_{\mathrm{T}}^{\mathrm{nom}}$ nominal areic dose for the transfer reference material

$D_{\mathrm{W}}^{\mathrm{nom}}$ nominal areic dose for the working reference material

D^{rec} received areic dose

D^{ret} retained areic dose