TECHNICAL REPORT

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[Surface chemical analysis — Proposed](#page-6-0) procedure for certifying the retained areic [dose in a working reference material](#page-6-0) [produced by ion implantation](#page-6-0) Surface che

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Reference number ISO/TR 16268:2009(E)

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Understanding

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.

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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. International Organization for Standard Internation For Standard Internation International State Conservation Provided by International State of the analytical Deciment International Provident International Conservation Pr

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 $(VAMAS)[2]$

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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 ionimplanted 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 10¹⁶ atoms/cm² to 10¹³ atoms/cm² (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 ionimplanted 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 **Copyright International Organization or Organization** of the unnit in which the property value is expressed, where the certified value is accompanied by international or of the unit in which the property value is expresse

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 d*N* by d*A*, where d*N* 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 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 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 d*N*imp by d*A*, where d*N*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 d*A* and captured within the solid

 $D^{imp} = dN^{imp}/dA$

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 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 Copyright International Organization of Standardization for Standardization Constrained By under the Standardization of the Standardization or networking permitted through the solid captured within the solid applare shape

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 d*N*rec by d*A*, where d*N*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 d*A*

 D rec = d *N*_{tec} $/dA$

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 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 d*N*ret by d*A*, where d*N*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 d*A* and permanently retained within the solid

 $D^{\text{ret}} = dN^{\text{ret}}/dA$

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 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 NOIE 2 The geometric surface area retres to the areal measure of the projection of the usually micro-rough surface

NOTE 3 The retained areic dose is smaller than the implanted areic dose if some of the implanted particles

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

ISO/TR 16268:2009(E)

5 Concept and procedure

5.1 General information

A fundamental problem in surface chemical analysis of a given material is the calculation of the local concentration of the analyte from the intensity of the signal registered by the measuring instrument. Generally preferred is a calculation based on a modelling of the signal excitation and measuring processes. For the powerful and widely used surface-analytical technique of secondary-ion mass spectrometry (SIMS), quantification via modelling has, hitherto, proved to be impossible. Instead, reference materials of similar and known composition are used for establishing a quantitative relationship between signal intensity and analyte concentration, based on the similarity principle and the rule of proportionality.

There are problems associated with this analysis approach that have not as yet been satisfactorily solved. A major problem is that no commercial supplier is prepared to prepare and stock certified reference materials (CRMs) for the great variety of multi-component materials in use. The cost of certifying potential CRMs can only be justified if some minimum number can be sold at an affordable price. This market can be estimated with some certainty only for CRMs used in the routine quality control of industrially established processes. The materials developer, experimenting with ever-new compositions and requiring a one-off CRM for every composition, cannot be catered for under this practice.

A solution to this problem is described in this Technical Report that addresses both the need of the analyst and the commercial reality of the reference material business. The solution is based on a new concept and procedure[1] that is not based on the current practice in which all reference materials, including working reference materials (WoRMs), are prepared, certified, certificated and sold by a commercial supplier. Instead, the analyst is given the opportunity to accept responsibility for the preparation and certification of special WoRMs, and the commercial supplier is merely expected to stock a small range of generic primary reference materials (PrRMs) and to certify and sell secondary reference materials (SeRMs) in response to market demand (i.e. just-in-time). The analyst, in turn, certifies the WoRM against the SeRM. Service providers are expected to assist in the preparation and certification of the reference materials.

The complete scheme is outlined in Table 1.

Table 1 — Referencing scheme for measurement of a chemical element (analyte) in a host matrix (The specimen and the two reference materials WoRM and TrRM are within the area of responsibility of the analyst. This Technical Report is concerned with the certification of the TrRM and the WoRM.)

Interpretation of table

Row 5: The specified PrRM is kept by a commercial supplier of reference materials.

Row 4: The SeRM is sold by this supplier as a certified copy of the PrRM, after being referenced by the referencing method in row 4.

Row 1: The given analyte in a given specimen is quantified by comparative measurement against a WoRM by the referencing method in row 1.

Row 2: The WoRM is referenced against the SeRM in a two-step process via a TrRM and a combination of the referencing methods in rows 2 and 3. In the first step, the WoRM is referenced against the TrRM by the referencing method in row 2.

Row 3: The TrRM is then referenced against the SeRM by the referencing method in row 3 (i.e. the second step of the two-step process of referencing of the WoRM against the SeRM).

As shown in Table 1, all four reference materials in the chain PrRM \rightarrow SeRM \rightarrow TrRM \rightarrow WoRM are of wafer or disc format into which the analyte has been introduced by ion implantation. For reasons explained in Annex C, the analyte is a chemical element of atomic number larger than that of silicon. The reason for the choice of ion implantation for the manufacture of reference materials is that, in general, the process is fast, cheap, versatile and well controlled. The quantity of analyte is measured during implantation and can be uniformly spread over the wafer surface. The analyte is safely stored inside the wafer and the depth distribution is sufficiently well known. Further, the commercial supplier benefits from the fact that the expenditure for the certification of the PrRM is a once-only expenditure because the PrRM is neither sold to the analyst nor is it consumed in the certification of the SeRMs against the PrRMs. This situation is made possible by the use of wavelength-dispersive X-ray fluorescence spectrometry (WD/XFS) for certification. This non-invasive analytical technique leaves the PrRM undamaged and reusable. or orse format into which the analysis and Definibility of the manufacture of reference materials is that, in general, they coess is fast, the non-thermodication of the ecologic or ion implantation of the with Internation

This Technical Report describes the certification of the two reference materials WoRM and TrRM, which fall within the area of responsibility of the analyst. The role of the supplier is beyond the scope of this Technical Report.

The analyst starts with the acquisition of a suitable SeRM from a commercial supplier. At the time of preparation of this Technical Report, only four suitable PrRMs or SeRMs were available from two suppliers: three CRMs from the US National Institute of Standards and Technology (NIST)^[4] and one from the European Institute for Reference Materials and Measurements (IRMM)[5]. A suitable CRM for this application is one for which a certified areic dose is provided that is appropriate for comparative measurement by WD/XFS. If a suitable CRM is not available, the analyst must either fall back on non-certified SeRMs (if commercially available) or follow the suggestion made in Annex D on how to prepare a substitute SeRM that is linked to a CRM.

The analyst then proceeds with the following two steps, explained in 5.2 and 5.3, respectively.

5.2 Preparation of the working and transfer reference materials

The analyst should contact a service provider for the preparation by ion implantation of the WoRM and the associated TrRM. The service provider is given a host wafer for the WoRM and a silicon wafer for the TrRM, together with instructions on the implantation energy and the respective nominal areic doses of the desired analyte. These nominal areic doses are not to exceed the respective upper critical areic doses. At the same time, the nominal areic dose of the TrRM must be larger than the lower critical areic dose in order to remain within the measurement range of WD/XFS. There is no such restriction on the nominal areic dose of the WoRM; this dose can, if required for the analytical task, be several orders of magnitude lower than that of the TrRM. The implantation energy must be at least as high as the critical energy for the wafer material with the lowest atomic number (most often silicon). This condition is essential for keeping ion backscattering from the wafer at a negligible value, such that the received and the retained areic doses are practically equal within the quantitative ion-implantation regime.

The analyst monitors adherence to these specifications and, most importantly, records the nominal areic doses actually measured. The ratio of the nominal areic doses for the WoRM and TrRM is taken to be equal to the ratio of the retained areic doses.

5.3 Measurement of retained areic dose in the transfer reference material

The retained areic dose in the TrRM is measured by comparative analytical measurements using WD/XFS against the known retained areic dose in the SeRM. A qualified service provider could make this measurement.

NOTE The use of WD/XFS as a near-surface analytical technique (inclusive of the use of ion-implanted reference materials for certification) requires computational correction for different depth distributions of the analyte. This topic is discussed in Annex C.

The retained areic dose of the ion-implanted analyte in the WoRM is then obtained from the measurement of the retained areic dose of the TrRM and the ratio of the nominal areic doses of the WoRM and TrRM obtained in 5.2.

5.4 Compatibility of the working reference material and the surface-analytical method

Considered here is the use of SIMS as the surface-analytical method in which the WoRM is to be used. For analysis by SIMS, only the analyte in the freshly sputtered surface is measured at any particular time. In other words, the analyte signal is generated not in the undisturbed material but in a so-called altered layer at the instantaneous surface where any pre-existing physical and chemical structure has been largely destroyed by the incident energetic ions. Hence, provided that the matrices of the specimen and the WoRM are as similar as they are expected (by design) to be, analysis by SIMS converts the analyte in either of the two matrices into quasi-identical states, and the proportionality of signal to concentration is transferable between them. It is then not necessary to specify or determine the physical and chemical states of the matrices during the SIMS measurements.

The working reference material and the specimen material should be handled in the same way during the time from ion implantation to the SIMS measurements in order to minimize the possibility that different amounts or types of surface contamination on the wafers might affect the SIMS measurements.

An analysis similar to that given here for SIMS needs to be made if another surface-analysis technique is utilized for the comparative measurements.

6 Requirements

6.1 Reference materials

The PrRM, SeRM and TrRM are high-purity silicon wafers (host wafers) of nominally 50 mm diameter (or more), ion-implanted under non-channelling conditions to a lateral uniformity of uncertainty $\leq 1\%$ (95%) confidence level) with nominally one isotope of a chemical element (the analyte) with an atomic number larger than that of silicon to a retained areic dose (the certified property) between the lower and upper critical areic dose values (typically in the range from a low 10¹⁵ atoms/cm² value to a high 10¹⁶ atoms/cm² value).

The WoRM is a wafer of the material required by the analyst of nominally 50 mm diameter (or more), ionimplanted under non-channelling conditions to a lateral uniformity of uncertainty $\leq \pm 1$ % (95 % confidence level) with nominally one isotope of a chemical element as the analyte to a retained areic dose below the upper critical areic dose value (typically in the range from a low 10^{13} atoms/cm² value to a high 10^{16} atoms/cm² value).

6.2 Instrumentation requirements

6.2.1 Ion implanter

For preparation of the WoRM and the TrRM, an ion implanter of implantation energy > 300 keV and, preferably, high mass resolution (ideally $M/\Delta M \geqslant 500^{[3]}$) with the following features is required:

- a) stability of current, composition and energy of the ion beam such that the uncertainty of each does not exceed \pm 1 % (95 % confidence level);
- b) the ability to separate neutrals from the ion beam or to operate at a target-station pressure at the low 10−5 Pa level;
- c) lateral uniformity of the areic dose in compliance with the intended application of the WoRM (see 6.2.3, 6.4 and Annex B);
- d) wafer mounting with tilt (to avoid axial channelling) and twist (to avoid planar channelling) [for Si(100) wafers, 7° tilt and 30° twist is recommended];
- e) overscan arrangement of Faraday cup(s) at the target station;
- f) Faraday cup(s) designed to reject externally generated secondary charged particles (electrons and ions).

6.2.2 Wavelength-dispersive X-ray fluorescence spectrometer

For comparative measurements of the TrRM against the SeRM, a wavelength-dispersive X-ray fluorescence spectrometer with the following features is required:

- a) an X-ray tube (preferably with a rhodium anode) operating at a power ≥ 3 kW;
- b) specimen rotation;
- c) a mask for defining identical exposure windows on different RMs.

The mask should be free of the analyte and should not give rise to spectral interference or a high background at the signal X-ray line.

6.2.3 Electron microprobe

Scanning-pattern generators in commercial ion implanters are designed to minimize the non-uniformity of the retained areic dose to an extent given in the generator specifications. This is generally satisfactory for all broad-beam analytical applications of the WoRM. Only if narrow-beam analytical application is intended may it be necessary to check the lateral uniformity of the retained areic dose by means of an electron microprobe with a wavelength-dispersive X-ray spectrometer, either on the TrRM or another suitable test specimen.

6.3 Ion-implantation requirements

The WoRM and TrRM are prepared sequentially in the same ion implanter under identical implanter operating conditions meeting the following requirements:

- a) the critical energy is exceeded for each RM;
- b) the ion beam current is set so that the lower critical implantation time is exceeded for each RM while ensuring that the maximum implantation time remains acceptable;
- c) the upper critical areic dose value for 99 % retention is not exceeded for each RM.
- NOTE 1 The critical energy is taken from literature sources (such as Reference [6]).

NOTE 2 The lower critical implantation time is the minimum time required to ensure a uniform areic dose across the target wafer and Faraday cup(s). This time is about 15 s (varying with the scan frequency) and is usually the minimum implantation time for the WoRM. The implantation time for the TrRM is related to the implantation time for the WoRM in the same ratio as the nominal areic doses of the two. For example, if the nominal areic dose of the TrRM is larger than that of the WoRM by a factor of 1 000, then the minimum implantation time for the TrRM is 15 000 s (i.e. just over 4 h).

6.4 Uniformity requirement

As explained in 6.2.3, scanning-pattern generators in commercial ion implanters are designed to minimize non-uniformity of the retained areic dose to an extent given in the generator specifications. This is generally satisfactory for all broad-beam analytical applications of the WoRM. Only if narrow-beam analytical application is intended may it be necessary to check the point-to-point variation by means of an electron microprobe, either on the TrRM or another suitable test specimen. The electron beam is applied in the point mode at randomly chosen positions on the surface of the TrRM or test specimen, and measurements are made of X-rays emitted from the analyte with a wavelength-dispersive X-ray spectrometer. Sufficient X-ray photon counts must be collected for the uncertainty to be reduced to the desired level (see Annex E).

7 Certification

7.1 Working reference material against the transfer reference material

Under conditions of quantitative ion implantation, when the received areic dose is, for all practical purposes, equal to the retained areic dose, the retained areic doses of the WoRM and TrRM, $D_{\text{W}}^{\text{ret}}$ and $D_{\text{T}}^{\text{ret}}$, respectively, are related to the corresponding measured nominal areic dose values, $D_{\text{W}}^{\text{num}}$ and respectively, are related to the corresponding measured nominal areic dose values, $D_{\rm W}^{\rm nom}$ and $D_{\rm T}^{\rm nom}$ follows:

$$
D_{\rm W}^{\rm ret} / D_{\rm T}^{\rm ret} = D_{\rm W}^{\rm nom} / D_{\rm T}^{\rm nom} \tag{1}
$$

7.2 Transfer reference material against the secondary reference material

WD/XFS is used for comparative measurement of the TrRM against the SeRM. X-ray fluorescence signals of identical signal type from the analyte are produced by identical X-ray exposures from identical surface areas on the two RMs. Covering each RM with the same mask ensures the exposure of and emission from identical surface areas. Copyright International Organization For Copyright Internation for Copyright Internation For Standardization Provided by International Organization From ITS Not for The Copyright International Organization Provident Inter If the TrRM and the SeRM have been ion-implanted at the same implantation energy and at the same angle of incidence, the analyte depth distributions will be identical and no attenuation correction is required. If either the implantation energy and the angle of incidence were different, or if both were different, then the analyte depth distributions will also be different in the two RMs, and the intensities of the X-ray fluorescence signals must be corrected for different attenuations (see Annex C and Reference [1]). No correction is required for inter-element excitation because the silicon host material is supposed to be implanted only with an analyte element with an atomic number higher than that of silicon (see Annex C and Reference [1]).

The retained areic doses of the TrRM and the SeRM, D_T^{ret} and D_S^{ret} , respectively, are then related to the corresponding measured, and if necessary corrected, X-ray fluorescence signals, $\mathcal{Q}_{\sf T}$ and $\mathcal{Q}_{\sf S}$, as follows:

$$
D_{\rm T}^{\rm ret} / D_{\rm S}^{\rm ret} = Q_{\rm T} / Q_{\rm S} \tag{2}
$$

7.3 Retained areic dose of the working reference material

The retained areic dose of the SeRM is known from its certificate. The retained areic dose of the TrRM can then be calculated from Equation (2). With the nominal areic doses known, the retained areic dose of the working reference material can then be obtained from Equation (1). For the uncertainties associated with these areic dose values, consult Annex E.

Annex A

(informative)

Ion implantation

Ion implantation is a particularly simple, fast and low-cost means of injecting a quantity (the nominal areic dose) of an analyte in a controlled way into the near-surface region of a host wafer. This deposition protects the analyte against physical handling of the host. The depth distribution of the ion-implanted analyte resembles a (usually skewed) bell profile, the centroid of which scales approximately linearly with the energy of the incident ions and is, for sub-MeV energies, in most cases in the sub-µm range. For many materials, a reasonable approximation of the analyte depth distribution can be derived from published data or by Monte Carlo simulation. These data and simulations normally pertain to depth distributions for small received areic doses when sputtering of the host material is still negligible.

Sputtering of the host material changes the depth distribution and contributes to a fractional loss of the implanted areic dose. This loss is negligible at small received areic doses; it becomes excessive when the sputtered thickness of the host becomes comparable to the distance of ion penetration into the host. Sputtering results in a difference between the implanted areic dose and the retained areic dose. Critical areic dose values have been calculated below which the difference remains insignificant^[1]. For instance, for 300 keV ion energy and perpendicular ion incidence, the upper critical areic doses (for 1 % difference between the implanted areic dose and the retained areic dose) in a host wafer of silicon range from 1.5×10^{18} atoms/cm² for P (atomic number 15) to 5×10^{16} atoms/cm² for Bi (atomic number 83).

Backscattering of incident ions from the target wafer is common and may be significant at low ion energies and for ions of low atomic mass. Backscattering results in a difference between the received areic dose and the implanted areic dose and can be reduced to insignificance by raising the ion energy to a sufficiently high value, called the critical energy in this Technical Report. The critical energy is strongly dependent on the ratio of the atomic mass of the analyte ion to the atomic mass of the host material and is well below 300 keV for an atomic-mass ratio of ≥ 1 . Values of critical energies are available^{[1][6]}. and Tor ions of low atomic mass. Backscattering results in a difference by texting the recorded area of ose and Copyright Internation Copyright Internation Provided area (as to reproduce the internation Provided area (as t

Annex B

(informative)

Ion-implantation dosimetry

At the down-stream end of commercial ion implanters, one or more Faraday cups, each electrically connected to a coulomb meter (current integrator), are used for ion-implantation dosimetry. A pencil-like ion beam of the analyte is scanned uniformly over both the cup aperture and the target wafer (in a so-called overscan arrangement). The nominal areic dose is derived from conversion of the charge deposited by the scanning ion beam in the Faraday cup to the number of ions incident per unit area of the cup aperture. In implantation technology, this nominal areic dose is taken to equal the received areic dose. For the purposes of this Technical Report, however, one must distinguish between the two and be aware of the various distinguishing factors (as pointed out in the main text).

Ideally, a Faraday cup should be like a "black body" with regard to charged particles. In other words, the cup should be open to charged particles moving from outside to the inside, but closed to charged particles of any type in the reverse direction. Furthermore, the Faraday cup should have the capability of rejecting any external secondary electrons and secondary ions. Not all Faraday cups in commercial implanters meet these requirements, and in such cases the mathematical conversion from measured charge to number of incident ions is inaccurate, giving rise to a systematic error. Another source of systematic error can be the ion beam itself, viz. if it transports ions other than $+1$ -charged ions of the analyte^[3]. In consequence, the nominal areic dose derived from the reading on the ion dosimeter may be only an approximate measure of the received areic dose.

Furthermore, a point-to-point variation of the received areic dose measured by ion dosimetry is likely to arise because the scanning of the pencil-like beam of ions across the target wafer may not be entirely uniform, although scanning-pattern generators are designed to minimize non-uniformity. Any such non-uniformity, if it exists, can be ignored for broad-beam analytical applications of the ion-implanted reference material so generated. For narrow-beam analytical applications, however, information must be obtained about any possible non-uniformity. The specifications of the scanning-pattern generator are one source of information, point-to-point analysis (of the TrRM or other test specimen) by electron microprobe is another.

Despite the significant dependence of the nominal areic dose on both the Faraday cup and the implanter operating conditions, nominal areic doses can be reproduced with high precision on successively implanted identical target wafers if the same Faraday cup is used under invariant operating conditions (as evidenced in the routine manufacture of semiconductor devices). In the case of the co-production of the WoRM and TrRM, the target wafers are usually not identical. Therefore, they emit, for a given ion beam intensity, significantly different numbers of secondary charged particles (electrons and ions). In order to keep the ratio of the nominal areic dose to the received areic dose unaffected by a change from one type of wafer to another, the Faraday cup must be so designed that all externally generated secondary charged particles are rejected at the cup entrance. requirements, and in such cases the mathematical convertisor is inaccurate, giving rise to a systematic form in the standard or the standard dose dose. Two Internation Provided by Internation Provided by Internation of the

Annex C

(informative)

X-ray fluorescence spectrometry

Comparative WD/XFS, the proposed means for referencing the TrRM against the SeRM, is not the only analytical technique suitable for referencing one retained areic dose against another. But WD/XFS stands out because of its good sensitivity, high specificity, wide dynamic range, easy mathematical treatment and wide availability. Furthermore, the (wholly inorganic) RMs are not degraded by extended or repeated exposure to X-radiation, as they would be in nuclear activation and from electron- or ion-beam exposure.

With available tube-sourced WD/XFS spectrometers used to characterize analytes implanted in a 50-mmdiameter wafer, retained areic doses greater than 1×10^{15} atoms/cm² of chemical elements of the third and higher periods of the periodic table can be measured with an uncertainty of \leq ±1 % (i.e. square-root uncertainty, see Annex E). Hence, 1×10^{15} atoms/cm² can be used as a lower critical areic dose for these elements. This value provides a convenient range to the upper critical areic dose (see Annex A) within which certification can be undertaken.

To avoid the problem of inter-element excitation that will occur when analyte elements of atomic number lower than silicon are implanted into silicon wafers, it is recommended that these analytes be excluded from the certification procedure described here. If one nevertheless wishes to certify the retained areic doses of these low-atomic-number analytes, one must apply a correction for inter-element excitation^[1]. Then, one should also be aware of the deterioration of the sensitivity of WD/XFS when applied to elements of the second period of the periodic table. As a result, the lower critical areic dose quickly rises with decreasing atomic number to values well above 1×10^{15} atoms/cm². Nevertheless, for most elements of the second period, there remains an open range to the upper critical areic dose which can be used for certification.

The X-ray fluorescence intensities measured on two RMs (here TrRM against SeRM) have to be related to their retained areic doses. The fluorescence X-rays (the excited radiation) are generated in the analyte as a result of irradiation by the exciting radiation (primary X-rays). The primary X-rays are exponentially attenuated in the host along their path towards the analyte, and the fluorescence X-rays are also exponentially attenuated in the host along their path away from the analyte towards the detector. This relatively simple situation is complicated only when the depth distributions of the analyte in the two RMs differ significantly from one another. Then, a correction must be applied which takes the differing depth distributions into account. The mathematics for this correction, called an attenuation correction, can be taken directly from Reference [1]. The attenuation correction formula in Reference [1] is exact for non-variant primary irradiation and if inter-element excitation is absent. It is to be noted that the attenuation correction formula in Reference [1] can correct not only for different ion-implanted profiles, but also for any depth distribution profile that can be expressed in terms of distribution moments.

Annex D

(informative)

Non-certified secondary reference materials and substitutes

Without suitable certified secondary reference materials (SeRMs) from trusted suppliers, the analyst has little choice but to use non-certified substitutes. Here, an approach is proposed based on the availability of at least a few certified reference materials (CRMs), in the form of silicon wafers, from suppliers such as the U.S. National Institute of Standards and Technology (NIST)^[4] and the European Institute for Reference Materials and Measurements (IRMM)^[5], whereby the analyst can generate a substitute for the non-available certified SeRM.

The key feature of the approach is that the analyte available in the CRM is ion-implanted into a virgin silicon wafer and that the relationship is established between the nominal areic dose and the retained areic dose in that wafer for the specific ion implanter and the specific implantation conditions. This relationship is expressed as a calibration factor (F_C) for the ion implanter used, for the implantation conditions used and for the particular analyte implanted to a nominal areic dose within the quantitative ion-implantation regime. The nominal areic dose is measured during the implantation process (see Annex B), and the retained areic dose is measured after implantation by comparative WD/XFS against the CRM (see Annex C). The calibration factor is then calculated as follows:

$$
F_{\mathbf{C}} = D^{\mathsf{ret}} / D^{\mathsf{nom}} \tag{D.1}
$$

The certification of the retained areic dose in the silicon wafer against that in the CRM turns the ion-implanted wafer into a genuine SeRM, of course. And the analyst can proceed to generate more SeRMs in the same way, even though they are not certified by a certifying authority. The limitation to this lies in the fact that currently only three CRMs are available which are suitable for the type of certification proposed here, viz. P in Si (NIST SRM 2133), As in Si (NIST SRM 2134) and Sb in Si (IRMM-302). Hence, the self-certified SeRMs are limited to the analytes P, As and Sb.

If another analyte is required, one can generate a reference material by ion-implanting the other analyte into a silicon wafer on the not unreasonable assumption that the calibration factor F_C is transferable to this other analyte, provided the ion implanter is the same, the implantation conditions are the same and the other analyte is implanted to a nominal areic dose within the quantitative ion-implantation regime. A prerequisite for the transferability of the F_C is that the implantation end of the ion implanter is not altered in any way that might affect the measurement of the nominal areic dose. Thus, the main factors affecting the transferability are the ionic and isotopic purity of the analyte ion beam^{[3][7]} and backscattering from the host wafer. The latter is estimated from published data^{[3][1]}, the former from the implantation conditions.

Annex E

(informative)

Uncertainties in measurements of areic dose

Uncertainty is a parameter of measurement that characterizes the dispersion of measured values of a measurand, here that of the (retained) areic dose. Uncertainty generally comprises several uncertainty components, arrived at by either Type A evaluation or Type B evaluation (see ISO/IEC Guide 98-3[8]). Type A evaluation of the uncertainty proceeds by statistical analysis of series of observations, Type B by means other than statistical analysis of series of observations. There are several uncertainty components to be considered for certification of the WoRM, arising from the succession of measurements from PrRM to WoRM. Typical values of uncertainty components are stated below, all of which were derived by Type B evaluation.

Every uncertainty component must be accompanied by a stated level of confidence, arrived at on the basis of the probability density function describing the probability of occurrence of measured values of the measurand. In common use are two probability density functions: the normal distribution if the measurand is a continuous random variable and the Poisson distribution if the measurand is a discrete random variable, like the X-ray photon count in WD/XFS. Either distribution predicts most measured values of the measurand to lie close to a mean value μ and ever fewer to lie farther away from the mean. The random nature of the variables causes about half of the measured values to lie above the mean and the other half below. An uncertainty interval ±*x*, denoting the range of measured values from $\mu - x$ to $\mu + x$, brackets a certain fraction of these measured values, but never all (because measured values outside any finite range are theoretically possible, though increasingly improbable). The fraction of measured values bracketed by the stated uncertainty interval is known as the level of confidence of this uncertainty interval. For a normal distribution, it is common practice to state one of three level-of-confidence values, viz. 68 %, 95 % and 99,7 %, associated with the uncertainty intervals $\pm\sigma$, $\pm 2\sigma$ and $\pm 3\sigma$, respectively, where σ is the standard deviation. Two important characteristics of the Poisson distribution are, firstly, that the standard deviation σ equals the square root of the mean μ and, secondly, that the Poisson distribution can be approximated by a normal distribution. Therefore, for a mean X-ray photon count of *N*, the standard deviation equals √*N*, and the uncertainty intervals ±√*N* and ±2√*N* are associated with level-of-confidence values of 68 % and 95 % (as for normal distributions).

During the research for this Technical Report, it was, for practical reasons, not possible to determine the uncertainty components by direct Type A evaluation. For measurements of X-ray photon counts in WD/XFS, a Poisson distribution was assumed for the dispersion of measured values, and the uncertainty intervals stated below all correspond to the measured-value range $N - \sqrt{N}$ to $N + \sqrt{N}$ and a level-of-confidence value of 68 %. All other measurands were assumed to be measured as analogue signals of the continuous-random-variable type, for which a normal distribution is representative of the dispersion of the measured values. The uncertainty intervals stated below were not derived from own measurements, however, but were copied from instrument specifications and published data from equivalent research and were not accompanied by level-ofconfidence values. Despite the non-specificity of these uncertainty intervals, they can be assumed to correspond to a level of confidence within the range 68 % to 95 %.

As a reminder, the areic dose as referred to here is not the point-to-point areic dose within the ion-implanted area of the reference material surface; rather, it is an average value obtained by dividing the integral dose received by the Faraday cup by the areal size of the cup aperture. See Annex B for further information.

The propagation of uncertainty components of the (average retained) areic dose is shown schematically in Table E.1 for a measurement sequence from PrRM to WoRM. For the stated uncertainties, a level of confidence of at least 68 % has been assumed.

For the purposes of this Technical Report, only the measurement sequence from SeRM to WoRM is of interest. That is, uncertainty U_S is assumed to be given and uncertainty components U_{T1} , U_{T2} , etc., and U_{W1} , *U_{W2}*, etc., have to be determined. Only the major uncertainty components are considered here, viz. those which are believed to make the largest contributions to the combined uncertainty U_W . These uncertainty components are specified in Tables E.2 and E.3. Reference [1] can be consulted for further information.

Reference material and typical uncertainty	Sequential cascade of uncertainties of the measurand areic dose			
PrRM	$U_P = \sqrt{(U_{P1}^2 + U_{P2}^2 + U_{P3}^2 +)}$			
\pm 1,0 % ($U_{\rm P}$)	where U_{P1} , U_{P2} , U_{P3} , etc., are uncertainty components of the measurand of the PrRM			
SeRM	$U_S = \sqrt{(U_P^2 + U_{S1}^2 + U_{S2}^2 +)}$			
\pm 1,5 % (U _S)	where U_{S1} , U_{S2} , etc., are uncertainty components of the measurand of the SeRM			
TrRM	$U_T = \sqrt{(U_S^2 + U_{T1}^2 + U_{T2}^2 +)}$			
\pm 1.8 % (U _T)	where U_{T1} , U_{T2} , etc., are uncertainty components of the measurand of the TrRM			
WoRM	$U_W = \sqrt{(U_T^2 + U_{W1}^2 + U_{W2}^2 +)}$			
± 2.0 % (U_W)	where U_{W1} , U_{W2} , etc., are uncertainty components of the measurand of the WoRM			

Table E.2

NOTE 1 The causes of the uncertainty components U_{T1} and U_{T2} are discussed in Reference [1], and an equation is proposed for their evaluation. The uncertainty is calculated as the square root of the X-ray photon count, i.e. the associated level of confidence is 68 %. The typical value given here is that for a signal-to-background ratio in excess of 100. It increases to \pm 0,65 % for a signal-tobackground ratio of 10. For the latter case, the typical value can be reduced to \pm 0,15 % by increasing the signal count to 10⁶.

NOTE 2 The uncertainty component U_{T3} arises from different attenuations of the exciting and the excited X-rays if the depth distributions of the analyte in the TrRM and SeRM happen to differ to a significant extent (as determined by actual analysis or by computer simulation). These different X-ray attenuations can be corrected for by application of Equation (1) in Reference [1]. There is an uncertainty in this correction as a result of uncertainties in the tabulated values of "mass attenuation coefficients" (MACs) of the exciting and the excited X-rays. The uncertainty in the correction can be worked out by means of Equation (1) in Reference [1] and equals *U*T3. The uncertainty vanishes if the depth distributions of the analyte are identical in the TrRM and SeRM. This can be achieved by arranging the TrRM to be ion-implanted at the same ion energy and to the same areic dose as the SeRM. NOTE 2 The uncertainty component U_{T3} arises from different attenuations of the exciting and the excited X-rays if the depth distributions of the analytic international SeRM happen to differ to a significant extent (as

Measurand	Uncertainty components	Cause	Method of determination	Typical values of uncertainty
Areic dose D_{M}	Uncertainty of areic dose of TrRM, U_T	Not applicable		± 1.8 %
of WoRM as derived by means of Equation (1)]				(68 % confidence)
	Uncertainty of D_{W}^{nom} in Equation (1), U_{W1}	Variation of	In situ Faraday cup calibration (see Note 1)	$< \pm 0.1 \%$.
		Faraday cup leakage		nil for a perfect Faraday cup
	Uncertainty of D_{W}^{nom} in Equation (1), U_{W2}	Integrator uncertainty (see Note 2)	Integrator specifications or calibration	± 0.1 %
	Uncertainty of D_T^{nom} in Equation (1), U_{W3}	Variation of	In situ Faraday cup calibration (see Note 1)	$< \pm 0.1 \%$.
		Faraday cup leakage		nil for a perfect Faraday cup
	Uncertainty of D_T^{nom} in Equation (1), U_{WA}	Integrator uncertainty (see Note 2)	Integrator specifications or calibration	± 0,1%

Table E.3

NOTE 1 In general, all three, the host material of the WoRM, the areic dose and the depth distribution of the ion-implanted analyte, are different from those of the TrRM. These differences have no effect on ion dosimetry if the Faraday cup is well designed, i.e. provided the cup cannot be entered by secondary charged particles (secondary ions and electrons) generated by incidence of the ion beam away from the cup aperture. This has to be demonstrated by measurement, in which the cup has to show zero current for all points of incidence of the ion beam away from the cup aperture. If not, the "leaking" cup should be replaced. A less desirable alternative is to determine the "cup leakage" for all implantation conditions and to use the appropriate leakage value as a correction. Uncertainty components U_{W1} and U_{W3} arise from any variations in this correction, not from the correction itself. In summary, U_{W1} and U_{W3} either vanish or are very small. Type A evaluation is possible.

NOTE 2¹ Uncertainty components U_{W2} and U_{W4} arise from the limited reproducibility of measurements of the current integrator attached to the Faraday cup. Typical specified uncertainties are about ± 0,1 % (68 % confidence level), but can be better. Type A evaluation is possible. NOTE 2 Uncertainty components U_{NC} and U_{NC} and U_{NC} and Standard term the limited reproduction is possible.

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NOTE The deprecated term "dose density" is used in this paper instead of the more recent ISO-accepted term "areic dose".

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NOTE 1 The deprecated term "dose density" is used in this paper instead of the more recent ISO-accepted term "areic dose".

NOTE 2 Attention is drawn to two corrections required on p. 562, viz. ug⋅cm⁻² instead of mg⋅cm⁻² in lines 15 and 25 from the bottom of the page.

- [4] <http://www.nist.gov/srm>
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