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Surface chemical analysis — X-ray photoelectron spectroscopy — Measurement of silicon oxide thickness

Analyse chimique des surfaces — Spectroscopie de photoélectrons par rayons X — Mesurage de l'épaisseur d'oxyde de silicium

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Foreword

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *X-ray photoelectron spectroscopy*.

This second edition cancels and replaces the first edition (ISO 14701:2011), which has been technically revised.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The measurement of the thickness of silicon oxide at the surface of silicon wafers has been conducted in the past by many methods. These generally apply to oxide layers thicker than 20 nm. It is often important to measure thicknesses in the range below 10 nm, and this document addresses the range below 8 nm using X-ray photoelectron spectroscopy. Problems arise in measuring film thicknesses in this thickness range since, for a layer to bond well to the substrate, it must form strong inter-atomic bonds at the interface so that a monolayer or more of layer and substrate interfacial material exists there. This material would not necessarily be a thermodynamically stable bulk material. Additionally, if the layer is reactive, its outer surface might have reacted with the environment and so be changed between fabrication and measurement. For the particular case of silicon dioxide on silicon, at the interface there is approximately a monolayer of sub-oxides and, at the surface, adsorbed materials containing carbon, oxygen and probably hydrogen atoms. These effects lead to offsets for the thicknesses deduced from many methods that, although reliably measuring changes in thickness between one sample and another, have difficulty in defining an absolute thickness.

The procedures described in this document provide methods to measure the thickness with high accuracy (optimally 1 %) and also, more rapidly and simply, at lower accuracy (optimally 2 %). It could also form a basis for the measurement of many film thicknesses on substrates, but without considerable further work, the uncertainties will be undefined.

Surface chemical analysis — X-ray photoelectron spectroscopy — Measurement of silicon oxide thickness

1 Scope

This document specifies several methods for measuring the oxide thickness at the surfaces of (100) and (111) silicon wafers as an equivalent thickness of silicon dioxide when measured using X-ray photoelectron spectroscopy. It is only applicable to flat, polished samples and for instruments that incorporate an Al or Mg X-ray source, a sample stage that permits defined photoelectron emission angles and a spectrometer with an input lens that can be restricted to less than a 6° cone semi-angle. For thermal oxides in the range 1 nm to 8 nm thickness, using the best method described in this document, uncertainties, at a 95 % confidence level, could typically be around 2 % and around 1 % at optimum. A simpler method is also given with slightly poorer, but often adequate, uncertainties.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Abbreviated terms and symbols

4.1 Abbreviated terms

HPLC	high-performance liquid chromatography
IPA	isopropyl alcohol

4.2 Symbols

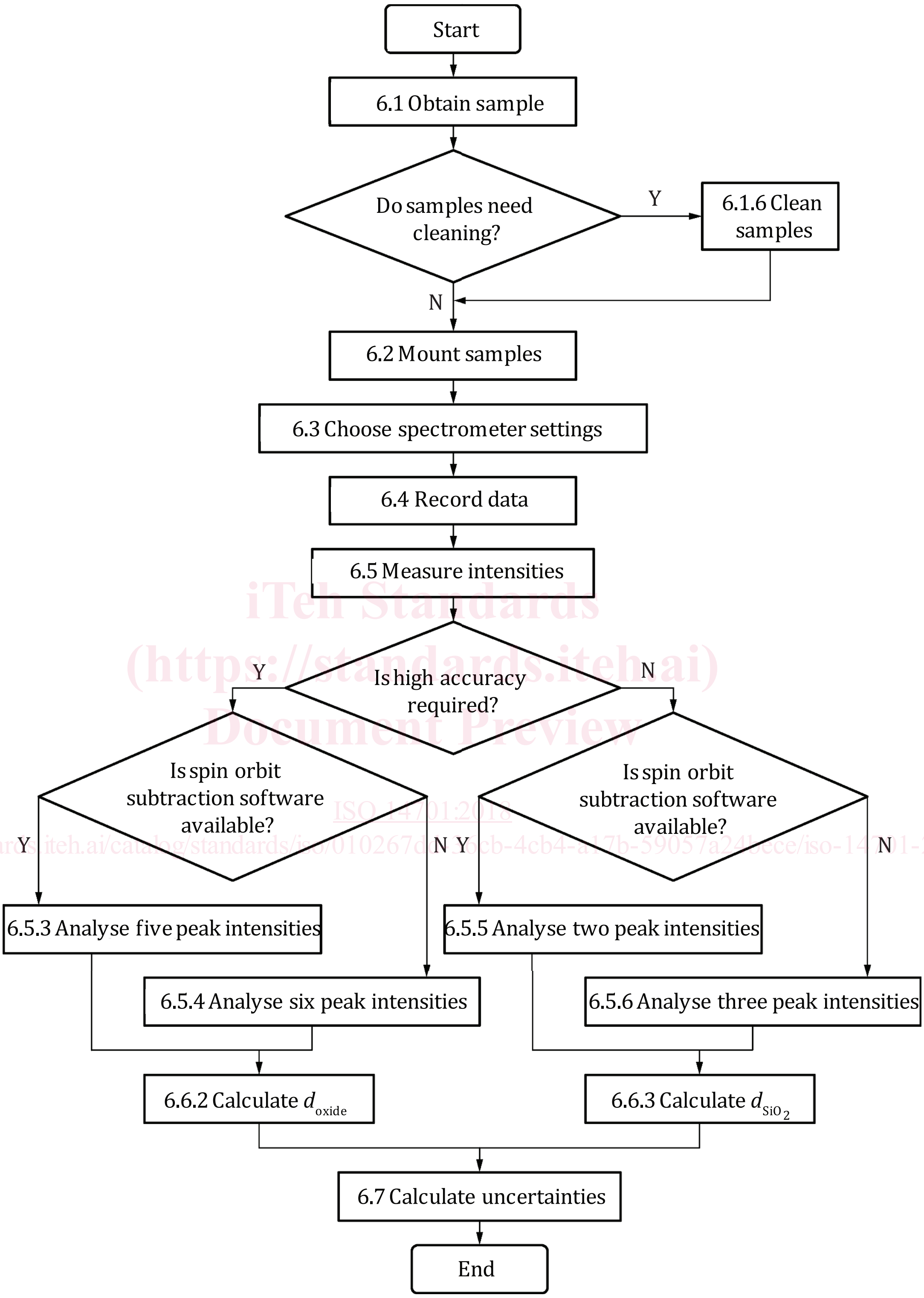
d_{oxide}	total oxide thickness
$d_{\mathrm{Si_2O}}$	thickness contribution to the $\mathrm{Si_2O}$ peak
d_{SiO}	thickness contribution to the SiO peak
$d_{\mathrm{Si_2O_3}}$	thickness contribution to the $\mathrm{Si_2O_3}$ peak

d_{SiO_2}	thickness contribution to the SiO_2 peak
I_{Si}	intensity of the Si contribution to the Si 2p peak
I_{Si_2O}	intensity of the Si_2O contribution to the Si 2p peak
I_{SiO}	intensity of the SiO contribution to the Si 2p peak
$I_{Si_2O_3}$	intensity of the Si_2O_3 contribution to the Si 2p peak
I_{SiO_2}	intensity of the SiO_2 contribution to the Si 2p peak
L_{Si}	attenuation length for Si 2p electrons in Si
L_{Si_2O}	attenuation length for Si 2p electrons in Si_2O
L_{SiO}	attenuation length for Si 2p electrons in SiO
$L_{Si_2O_3}$	attenuation length for Si 2p electrons in Si_2O_3
L_{SiO_2}	attenuation length for Si 2p electrons in SiO_2
R_{Si_2O}	intensity normalization parameter for the Si_2O contribution to the Si 2p peak
R_{SiO}	intensity normalization parameter for the SiO contribution to the Si 2p peak
$R_{Si_2O_3}$	intensity normalization parameter for the Si_2O_3 contribution to the Si 2p peak
R_{SiO_2}	intensity normalization parameter for the SiO_2 contribution to the Si 2p peak
U_n	uncertainty contribution, at a 95 % confidence level, for the spectrum measurement statistics
U_θ	uncertainty contribution, at a 95 % confidence level, for θ
U_A	uncertainty contribution, at a 95 % confidence level, for the analyser electron optics defining the solid angle of acceptance
U_E	uncertainty contribution, at a 95 % confidence level, for the validity of the equations for thicknesses
U_F	uncertainty contribution, at a 95 % confidence level, for peak synthesis without the intermediate oxides
U_L	uncertainty contribution, at a 95 % confidence level, for the attenuation length
θ	angle of emission of electrons measured from the surface normal

5 Outline of method

Here, the method is outlined so that the detailed procedure, given in Clause 6, can be understood in context. Typical spectra are available in the literature and given later in Figures 3 and 4.

The initial step of cleaning the samples, if necessary, is given in 6.1. In 6.2 and 6.3, the samples are mounted and suitable spectrometer settings chosen. In 6.4 and 6.5, the procedures for recording the data and measuring the intensities are given. Finally, in 6.6 and 6.7, the oxide thickness and its uncertainty at a confidence level of 95 % are calculated. In 6.5 and 6.6, two methods are provided for calculating the oxide thicknesses from the data: a more complex method with better uncertainties and a simpler method with poorer uncertainties. The more complex method might achieve uncertainties as low as 1 %, but the simpler method is restricted to uncertainties that are greater than 2 %. This greater figure is often adequate for many purposes, however. The sequence of steps is illustrated in the flowchart in Figure 1. It might be useful to refer to this while using this document.



Key

Y	Yes
N	No

Figure 1 — Flowchart of the measurement process

Subclause 6.3 requires the angle of emission to be set accurately, and it is often the accuracy of this setting that limits the final accuracy. Users of this procedure will need to ensure that the accuracies of these settings are known in order to evaluate the final uncertainty. The settings can be checked to an adequate level using reflectors mounted on the sample stage, a laser beam and standard geometrical relationships[1][2].

6 Method for measuring the oxide thickness

6.1 Cleaning and preparing the sample

6.1.1 For cleaning and preparing the samples, gloves and uncoated stainless-steel tweezers are required. In selecting gloves, care shall be taken to avoid those with talc, silicone compounds or similar contaminants. “Powder-free” gloves have no talc, and fresh polyethylene gloves, or gloves of a higher quality, shall be used in sample handling. Do not use moulded gloves, for example vinyl, which will probably be covered with highly contaminating release agents. Tweezers that are of uncoated stainless steel shall be used.

6.1.2 To manipulate samples, the gloves are used to hold the tweezers and not the sample. Avoid any wiping materials, sometimes used to handle samples, as they might result in unwanted contamination of the sample surface. Unnecessary contact of the sample with the gloves shall be avoided. Sample mounts and other materials used to hold samples shall be cleaned regularly whenever there is a possibility of cross-contamination of samples. The use of tapes containing silicones and other mobile species shall be avoided[3].

6.1.3 Samples shall be prepared and mounted with clean tweezers to ensure that the surface is not altered prior to analysis and that the best possible vacuum conditions are maintained in the analytical chamber. Use the gloves to handle the tweezers to avoid contaminating them or any cleaning equipment with finger grease. Clean the tweezers by one of the following two methods:

a) Immerse the tweezers before use for 16 h in electronic, or equivalent, grade (>99,9 %) isopropyl alcohol (IPA) that leaves no significant residue. If electronic grade IPA is not available, high-performance liquid chromatography (HPLC) grade (>99,5 %) IPA may be used.

b) Boil the tweezers in ultra-high-purity water for 10 min.

Grip the sample at the edge only, in a region that will not be analysed. Avoid breathing or speaking over the sample. Keep these tweezers in a clean glass container for future use. Tools shall not unnecessarily touch the sample surface to be analysed.

6.1.4 Inspect the samples for any scratches, blemishes or marks on the polished surfaces. Finger marks should not be present but, if they are, may be removed as described in 6.1.6. Note the condition of the surface. It should be featureless. Identify the side of the sample for analysis. This is usually the polished side. If the unpolished side is to be analysed, this document is not applicable. If the sample is too large for insertion into the instrument, a smaller portion will need to be cut from it. To do this, material with a (100)-orientated surface may be cut to form a suitably sized rectangular portion by cleavage along (111) planes. In this way, a square of side 10 mm, bounded by <110> directions, may be conveniently produced. For those samples with a (111)-orientated surface, a similar cleaving along (111) planes forms equilateral triangles, also bounded by <110> directions. Triangle sides of length 15 mm are convenient. The scribing for cleaving often leaves very small fragments of Si on the samples. These fragments shall, as far as possible, be removed. The cleaning procedure described in 6.1.6 is often sufficient for this purpose.

NOTE The <110> directions are usually indicated by flats cut into the sides of (100)- and (111)-orientated wafers.

6.1.5 Analyses show that wafers and many other materials, such as metals, accumulate organics, hydrocarbons, silanes and phthalates from the environment. During storage of wafers, the thickness