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

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National foreword

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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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**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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ISO 14701 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *X-ray photoelectron spectroscopy*.

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 International Standard 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, whilst reliably measuring changes in thickness between one specimen and another, have difficulty in defining an absolute thickness.

The procedures described in this International Standard 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 International Standard 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 specimens and for instruments that incorporate an Al or Mg X-ray source, a specimen 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 International Standard, 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 Symbols and abbreviations

2.1 Abbreviations

HPLC high-purity liquid chromatography

IPA isopropyl alcohol

2.2 Symbols

The term intensity is used below and elsewhere. This refers to a measurement of peak area in the spectrum after relevant background subtraction.

d_{oxide} total oxide thickness

$d_{\text{Si}_2\text{O}}$ thickness contribution to the Si_2O peak

d_{SiO} thickness contribution to the SiO peak

$d_{\text{Si}_2\text{O}_3}$ thickness contribution to the 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

$I_{\text{Si}_2\text{O}}$ intensity of the Si_2O contribution to the Si 2p peak

I_{SiO} intensity of the SiO contribution to the Si 2p peak

$I_{\text{Si}_2\text{O}_3}$ 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

$L_{\text{Si}_2\text{O}}$	attenuation length for Si 2p electrons in Si_2O
L_{SiO}	attenuation length for Si 2p electrons in SiO
$L_{\text{Si}_2\text{O}_3}$	attenuation length for Si 2p electrons in Si_2O_3
L_{SiO_2}	attenuation length for Si 2p electrons in SiO_2
$R_{\text{Si}_2\text{O}}$	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
$R_{\text{Si}_2\text{O}_3}$	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

3 Outline of method

Here, the method is outlined so that the detailed procedure, given in Clause 4, 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 specimens, if necessary, is given in 4.1. In 4.2 and 4.3, the specimens are mounted and suitable spectrometer settings chosen. In 4.4 and 4.5, the procedures for recording the data and measuring the intensities are given. Finally, in 4.6 and 4.7, the oxide thickness and its uncertainty at a confidence level of 95 % are calculated. In 4.5 and 4.6, 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 during use of this International Standard.