INTERNATIONAL STANDARD

ISO 14701

Second edition
2018-11

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

Reference number
ISO 14701:2018(E)

© ISO 2018



COPYRIGHT PROTECTED DOCUMENT

© ISO 2018

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Abbreviated terms and symbols **1**
4.1 Abbreviated terms 1
4.2 Symbols 1
5 Outline of method **2**
6 Method for measuring the oxide thickness **4**
6.1 Cleaning and preparing the sample 4
6.2 Mounting the sample 5
6.3 Choosing spectrometer settings 5
6.4 Recording data 8
6.5 Measuring intensities 9
6.6 Calculating the oxide thickness 12
6.7 Calculating the uncertainty of the oxide thickness 14
Bibliography **16**

© ISO 2018 – All rights reserved

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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.

© ISO 2018 – All rights reserved

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.

© ISO 2018 – All rights reserved

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_{Si_2O}	thickness contribution to the Si_2O peak
d_{SiO}	thickness contribution to the SiO peak
$d_{Si_2O_3}$	thickness contribution to the Si_2O_3 peak

© ISO 2018 – All rights reserved